

Supramolecular gating of TADF process in self-assembled nano-spheres for high-resolution OLED applications

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1. General procedures and instrumentation

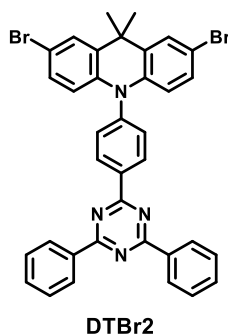
All the starting materials and solvents were purchased from commercial sources and used without further purification. ^1H and ^{13}C NMR spectra were collected on a 400 MHz spectrometer at room temperature. UV-visible absorption spectra were recorded on a HITACHI U2800A or Perkin-Elmer spectrophotometer. Steady-state fluorescence spectra were collected on a Horiba Fluoromax instrument. Decay parameters were extracted from reconvolution of the time-resolved emission decay profiles collected using a home-built TC-SPC instrument equipped with a Picoquant 310 nm or 350 nm pulsed excitation source and a Hamamatsu R6427 photomultiplier and Timeharp 260 collection electronics. A monochromator or 420 long-pass filter was used to isolate the emission from the sample. Variable-temperature spectra and lifetimes were obtained using an Oxford DN optistat connected to an ITC controller (precision = $\pm 0.1\text{K}$). Samples for emission experiments and quantum yields were degassed by four freeze-pump-thaw cycles and flame-sealed unless indicated otherwise.

The fluorescence microscope used was a Picoquant Microtime 200 based on an inverted Olympus IX71 microscope equipped with a $100\times$ objective (UPLSAPO, N.A. = 1.4), a PicoHarp 300 multichannel single photon counter, and two MPD SPADs. A 375 nm laser diode was used for PL measurements. The EL devices were placed flat on the sample tray and the set-up was used to collect the either the PL transmission through the dichroic optics before being focused on a $50\text{ }\mu\text{m}$ pinhole. After the pinhole, light was diverted into an Andor SR300i spectrometer equipped with a Newton EM-CCD for spectroscopy measurements or to time-resolved avalanche photodiodes (MPD). In a second mode (wide field), an epi-illumination was set with a 360 nm LED and direct imaging of the field of view on a sCMOS camera, after filtering the PL with an appropriate dichroic mirror and filters. With this last mode, EL could also be imaged on the camera.

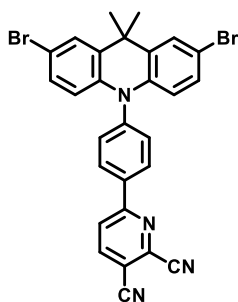
External quantum efficiency measurements: A 3D-printed device's compartment coupled, via optical fiber, to a UV-VIS spectrophotometer (Ocean Optics HR2000) was employed for the quantitative device's EQE analysis. The system was calibrated using a commercially available white light-emitting diode (Thorlabs, model LEDSW30), whose *optical power vs forward current* and *angular intensity distribution* data are provided by the supplier, aligned to the optical fiber entrance. A constant current of 0.1 mA was applied with a Power-Source-Unit (Keithley 2400) and the corresponding EL spectrum was registered during the integration time (50 ms); according to the *optical power/current* dependence, 0.1 mA yields an optical power of 0.072 mW, and therefore, the area below the EL spectrum was normalized to this value with the aim of converting from counts to optical power units. Upon calibration, the optical power spectra of our devices at different voltages were measured during the same integration time, while the

electrical current was monitored. Considering the different spectral distribution of the target devices compared to the reference LED, a conversion from power units to number of photons was carried out; subsequent integration was performed to obtain the absolute number of photons, and then divided by the electric charge that flowed during the integration time, to finally obtain the corresponding EQE values. The different angular distributions and active areas of both kind of samples were contemplated for a more accurate EQE estimation. The active areas of both kind of devices, reference and target, were always placed at the exactly same position/distance from the optical fiber entrance to ensure reliability.

2. Synthetic procedures

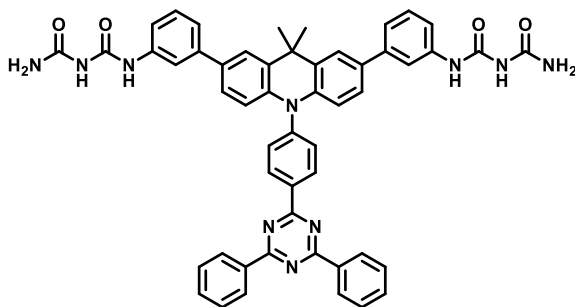


Synthesis of DTBr2. A mixture of **DMAC-TRZ** (500 mg, 0.97 mmol), NBS (758 mg, 4.26 mmol) was dissolved in THF and stirred at 0°C for 1 h and warmed to room temperature for 2 h. The reaction was quenched by adding saturated K₂CO₃ aqueous solution and extracted with CH₂Cl₂. The combined organic phase was washed with brine and dried over MgSO₄. The crude product was purified by column chromatography on silica gel (CH₂Cl₂/Hexane = 1/4) to afford pure **DTBr2** (76%, 496 mg) as a yellow solid. ¹H NMR (CDCl₃, 400 MHz) δ 9.03 (d, *J* = 3.40 Hz, 2H), 8.81 (d, *J* = 4.00 Hz, 4H), 7.67-7.59 (m, 6H), 7.56 (d, *J* = 1.20 Hz, 2H), 7.51 (d, *J* = 4.20 Hz, 2H), 7.11 (d, *J* = 1.00 Hz, 2H), 7.09 (d, *J* = 1.00 Hz, 2H), 6.26 (d, *J* = 4.40 Hz, 2H), 1.70 (s, 6H); ¹³C NMR (CDCl₃, 400 MHz) δ 171.9, 170.9, 144.3, 139.4, 136.7, 136.0, 132.7, 131.8, 131.7, 129.4, 129.0, 128.7, 128.2, 115.9, 113.6, 36.3, 31.0; HRMS (*m/z*, FAB⁺) Calcd for C₃₆H₂₆Br₂N₄ 674.4400; found 674.4268.



Py56Br2

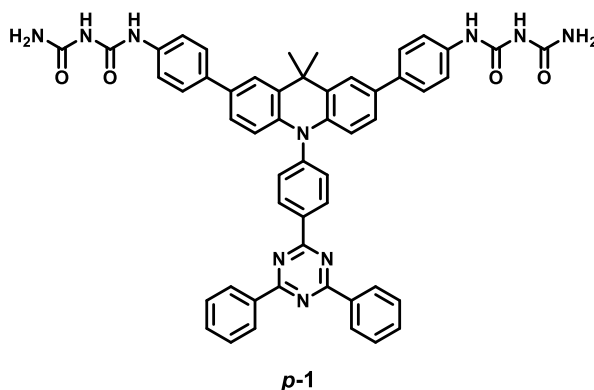
Synthesis of Py56Br2. A mixture of **Py56** (800 mg, 1.94 mmol), NBS (686 mg, 3.88 mmol) was dissolved in THF and stirred at 0°C for 1 h and warmed to room temperature for 2 h. The reaction was quenched by adding saturated K₂CO₃ aqueous solution and extracted with CH₂Cl₂. The combined organic phase was washed with brine and dried over MgSO₄. The crude product was purified by column chromatography on silica gel (CHCl₃/Hexane = 1/3) to afford pure **Py56Br2** (87%, 970 mg) as an orange solid. ¹H NMR (d₆-DMSO, 400 MHz) δ 8.75 (d, *J* = 8.5 Hz, 1H), 8.66 (d, *J* = 8.5 Hz, 1H), 8.51 (d, *J* = 8.5 Hz, 2H), 7.63 (dd, *J* = 11.8, 5.4 Hz, 4H), 7.19 (dd, *J* = 2.3 Hz, 2H), 6.15 (d, *J* = 8.8 Hz, 2H), 1.64 (s, 6H); ¹³C NMR (d₆-DMSO, 400 MHz) δ 158.67, 142.48, 138.74, 135.47, 135.45, 131.64, 131.37, 130.50, 129.29, 127.99, 123.89, 115.68, 114.89, 114.63, 112.68, 112.24, 35.85, 30.86; HRMS (*m/z*, FAB⁺) Calcd for C₂₈H₁₈Br₂N₄ 567.990; found 567.9974.



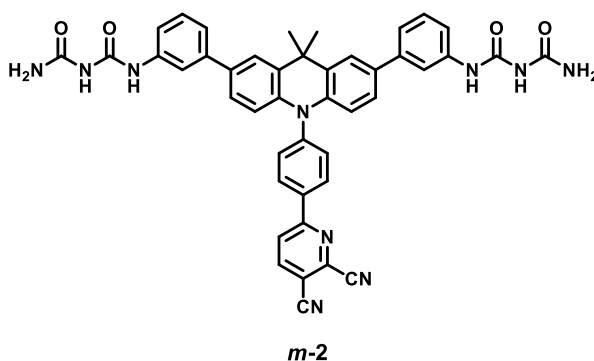
***m*-1**

Synthesis of *m*-1. A mixture of Pd(PPh₃)₄ (85 mg, 0.073 mmol), K₂CO₃ (615 mg, 4.45 mmol), **DTBr2** (500 mg, 0.74 mmol), 3-pinacolatoboronic ester-benzenebiuret (475 mg, 1.55 mmol) in degassed THF (7.5 mL) and water (2.2 mL) was refluxed for two days. The reaction was quenched by adding water and extracted with THF. The combined organic solution was washed with brine and dried over MgSO₄. The crude product was purified by column chromatography on silica gel (THF/CH₂Cl₂ = 1/1) to afford ***m*-1** (22%, 141 mg) as a yellow solid. ¹H NMR (d₆-DMSO, 400 MHz) δ 10.02 (s, 2H), 9.06 (d, *J* = 4.20 Hz, 2H), 8.86 (s, 2H), 8.79 (d, *J* = 3.80 Hz, 4H), 7.80 (s, 2H), 7.74-7.64 (m, 12H), 7.43 (d, *J* = 3.60 Hz, 2H), 7.36-7.30 (m, 6H), 6.90 (bs, 4H), 6.36

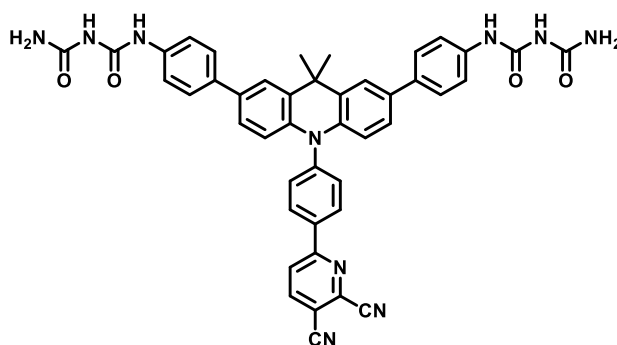
(d, $J = 4.00$ Hz, 2H), 1.82 (s, 6H); ^{13}C NMR (d_6 -DMSO, 400 MHz) δ 171.2, 170.5, 155.4, 152.1, 144.8, 140.9, 139.3, 138.6, 135.5, 135.3, 133.1, 132.7, 131.7, 131.5, 130.2, 129.4, 129.0, 128.7, 125.1, 124.2, 121.0, 117.5, 116.8, 114.4, 36.0, 32.0; HRMS (m/z , FAB $^+$) Calcd for $\text{C}_{52}\text{H}_{42}\text{N}_{10}\text{O}_4$ 870.3390; found 870.3322.



Synthesis of *p*-1. A mixture of $\text{Pd}(\text{PPh}_3)_4$ (68 mg, 0.059 mmol), K_2CO_3 (492 mg, 3.56 mmol), **DTBr2** (400 mg, 0.59 mmol), 4-pinacolatoboronic ester-benzenebiuret (380 mg, 1.24 mmol) in degassed THF (6 mL) and water (1.8 mL) was refluxed for two days. The reaction was quenched by adding water and extracted with THF. The combined organic solution was washed with brine and dried over MgSO_4 . The crude product was purified by column chromatography on silica gel ($\text{THF}/\text{CH}_2\text{Cl}_2 = 1/1$) to afford ***p*-1** (17%, 90 mg) as a yellow solid. ^1H NMR (d_6 -DMSO, 400 MHz) δ 10.01 (s, 2H), 9.05 (d, $J = 4.00$ Hz, 2H), 8.86 (s, 2H), 8.79 (d, $J = 3.60$ Hz, 4H), 7.80 (s, 2H), 7.74-7.67 (m, 8H), 7.60 (d, $J = 4.20$ Hz, 4H), 7.50 (d, $J = 4.20$ Hz, 4H), 7.31 (d, $J = 4.20$ Hz, 2H), 6.90 (bs, 4H), 6.34 (d, $J = 4.40$ Hz, 2H), 1.82 (s, 6H); ^{13}C NMR (d_6 -DMSO, 400 MHz) δ 171.2, 170.5, 155.4, 151.9, 144.9, 138.9, 136.9, 135.4, 135.3, 134.8, 133.1, 132.3, 131.6, 131.4, 131.3, 130.2, 129.0, 128.7, 128.6, 126.5, 124.6, 123.7, 119.4, 114.4, 36.0, 32.0; HRMS (m/z , FAB $^+$) Calcd for $\text{C}_{52}\text{H}_{42}\text{N}_{10}\text{O}_4$ 870.3390; found 870.3472.



Synthesis of *m*-2. A mixture of Pd(PPh₃)₄ (40 mg, 0.035 mmol), K₃PO₄ (447 mg, 2.10 mmol), **Py56Br2** (200 mg, 0.35 mmol), 3-pinacolatoboronic ester-benzenebiuret (225 mg, 0.74 mmol) in degassed THF (7.5 mL) and water (1.0 mL) was refluxed for two days. The reaction was quenched by adding water and extracted with THF. The combined organic solution was washed with brine and dried over MgSO₄. The crude product was purified by column chromatography on silica gel (THF/CH₂Cl₂ = 1/1) to afford ***m*-2** (12%, 33 mg) as an orange solid. ¹H NMR (d₆-DMSO, 400 MHz) δ 10.02 (s, 2H), 8.87 (s, 2H), 8.74 (d, *J* = 4.20 Hz, 1H), 8.66 (d, *J* = 4.20 Hz, 1H), 8.53 (d, *J* = 4.00 Hz, 2H), 7.78 (s, 2H), 7.67 (d, *J* = 4.20 Hz, 2H), 7.63 (s, 2H), 7.43 (d, *J* = 4.00 Hz, 2H), 7.38-7.28 (m, 6H), 6.90 (bs, 4H), 6.31 (d, *J* = 4.20 Hz, 2H), 1.80 (s, 6H); ¹³C NMR (d₆-DMSO, 400 MHz) δ 159.0, 155.4, 152.1, 143.4, 142.6, 141.0, 138.6, 135.6, 135.4, 132.7, 131.7, 130.5, 130.2, 129.3, 125.1, 124.1, 124.0, 121.0, 117.5, 116.8, 115.1, 114.8, 114.4, 112.3, 36.0, 31.8; HRMS (*m/z*, FAB⁺) Calcd for C₄₄H₃₄N₁₀O₄ 766.2764; found 766.2783.



***p*-2**

Synthesis of *p*-2. A mixture of Pd(PPh₃)₄ (40 mg, 0.035 mmol), K₃PO₄ (447 mg, 2.10 mmol), **Py56Br2** (200 mg, 0.35 mmol), 4-pinacolatoboronic ester-benzenebiuret 225 mg, 0.74 mmol) in degassed THF (7.5 mL) and water (1.0 mL) was refluxed for two days. The reaction was quenched by adding water and extracted with THF. The combined organic solution was washed with brine and dried over MgSO₄. The crude product was purified by column chromatography on silica gel (THF/CH₂Cl₂ = 1/1) to afford ***p*-2** (15%, 41 mg) as an orange solid. ¹H NMR (d₆-DMSO, 400 MHz) δ 10.01 (s, 2H), 8.86 (s, 2H), 8.74 (d, *J* = 4.20 Hz, 1H), 8.66 (d, *J* = 4.20 Hz, 1H), 8.52 (d, *J* = 4.20 Hz, 2H), 7.78 (s, 2H), 7.65 (d, *J* = 4.40 Hz, 2H), 7.59 (d, *J* = 4.40 Hz, 4H), 7.50 (d, *J* = 4.20 Hz, 4H), 7.29 (d, *J* = 3.40 Hz, 2H), 6.90 (bs, 4H), 6.28 (d, *J* = 4.20 Hz, 2H), 1.80 (s, 6H); ¹³C NMR (d₆-DMSO, 400 MHz) δ 159.0, 155.4, 151.9, 143.5, 142.6, 138.9, 136.9, 135.6, 135.3, 134.8, 132.3, 131.7, 130.5, 130.3, 126.5, 124.6, 124.0, 123.7, 119.4, 115.1, 114.8, 114.4, 112.3, 36.0, 31.8; HRMS (*m/z*, FAB⁺) Calcd for C₄₄H₃₄N₁₀O₄ 766.2764; found 766.2777.

3. Scanning electron microscopy

Samples were prepared by drop-casting a solution of **1** (0.1 mM) onto a flat SiO₂/Si substrate as described above. The samples were imaged using a FEI Nova NanoSEM 200 in low-vacuum mode with no conductive overcoat. The chamber pressure was maintained at 0.45 Torr water using a differential pumping system. An immersion lens was employed and the secondary electrons amplified by gas vapor and collected by an electrode mounted on the pole piece.

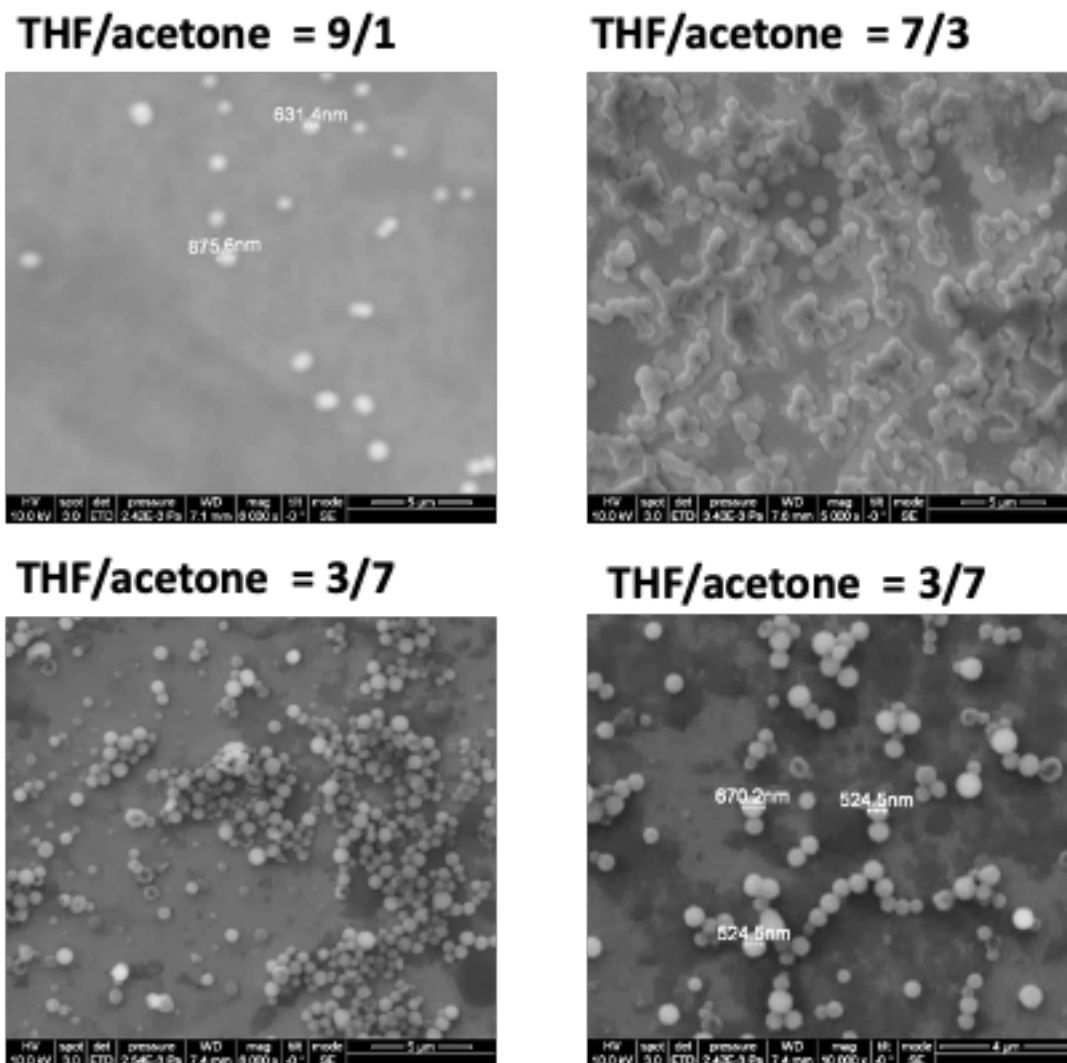
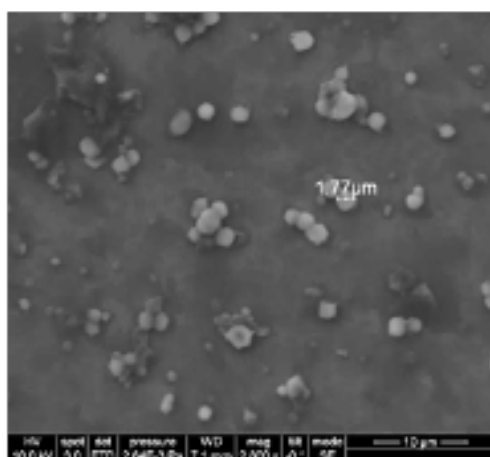
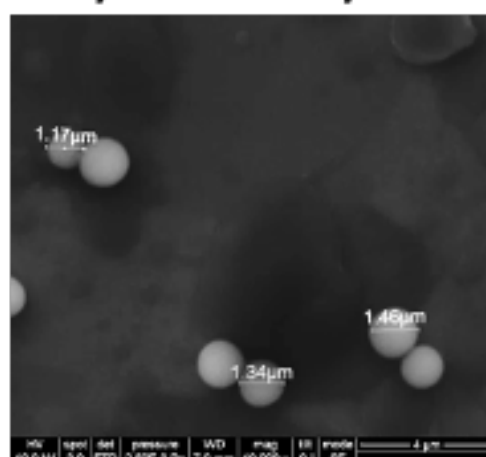


Figure S1. SEM images of **m-1** drop-cast from different solutions.

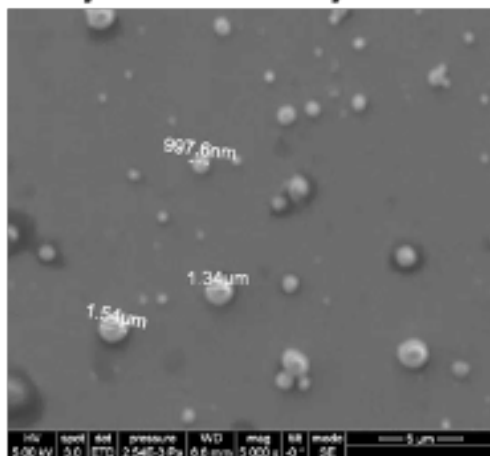
THF



THF/acetone = 7/3



THF/acetone = 1/1



THF/acetone = 1/9

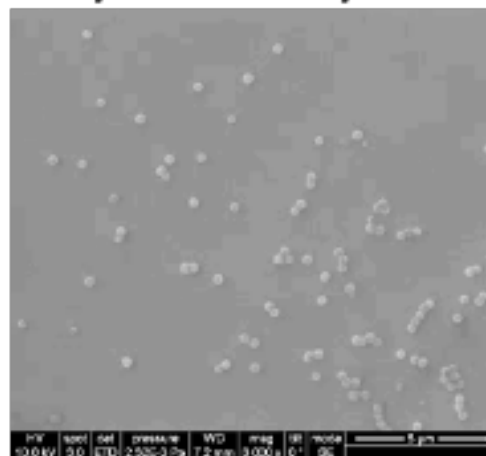
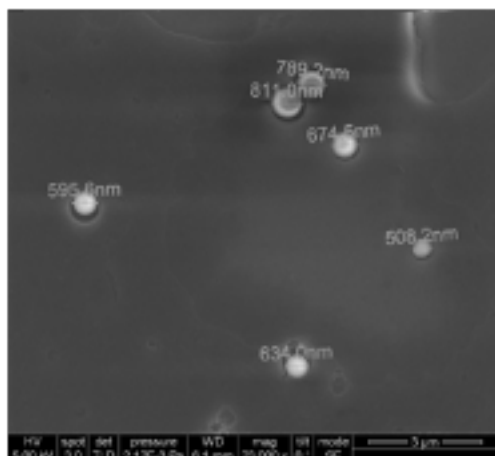
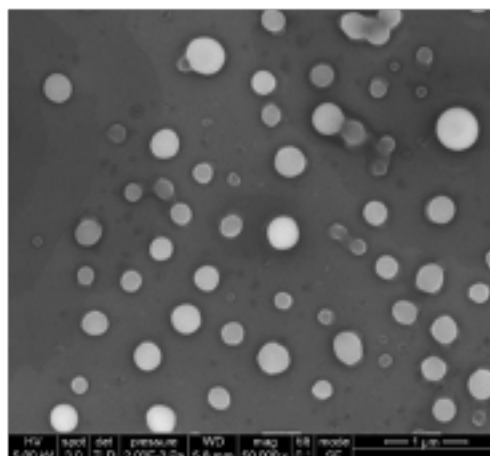


Figure S2. SEM images of *p*-1 drop-cast from different solutions.

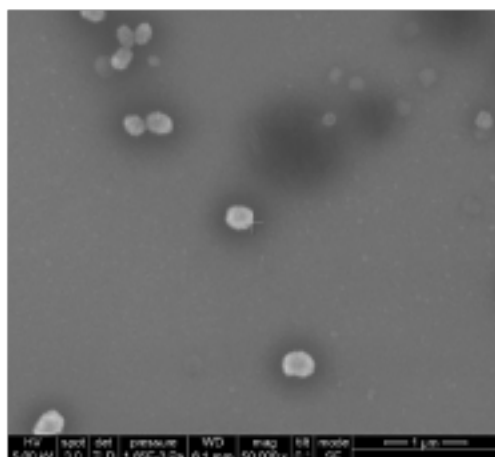
THF



THF/acetone = 1/1



THF/acetone = 3/7



THF/acetone = 1/9

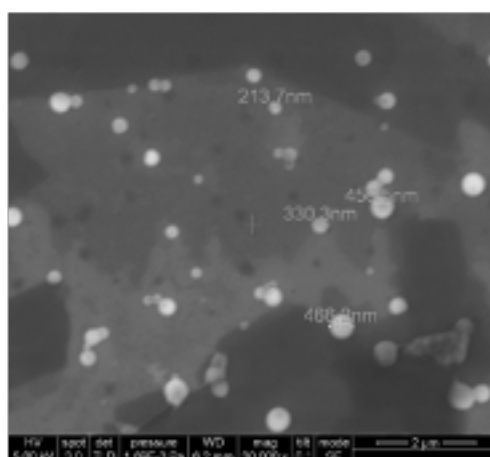


Figure S3. SEM images of *m-2* drop-cast from different solutions.

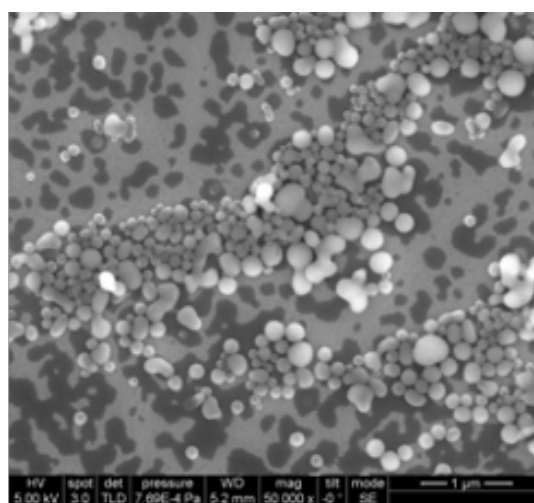
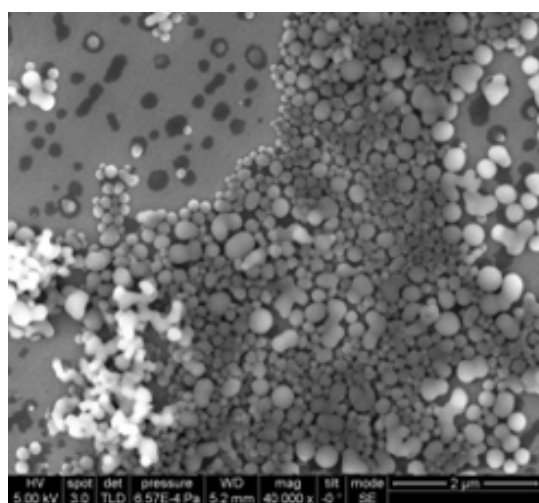


Figure S4. SEM images of *p-2* drop-cast from different solutions.

4. Transmission electron microscopy

Samples were drop-casted onto a 200-mesh copper grid coated with formvar film stabilized with vacuum-evaporated carbon and dried under air. The samples were examined in electron microscopes operating at 75 kv (Hitachi H-7650) and at 200 kv (JEOL JEM-2100).

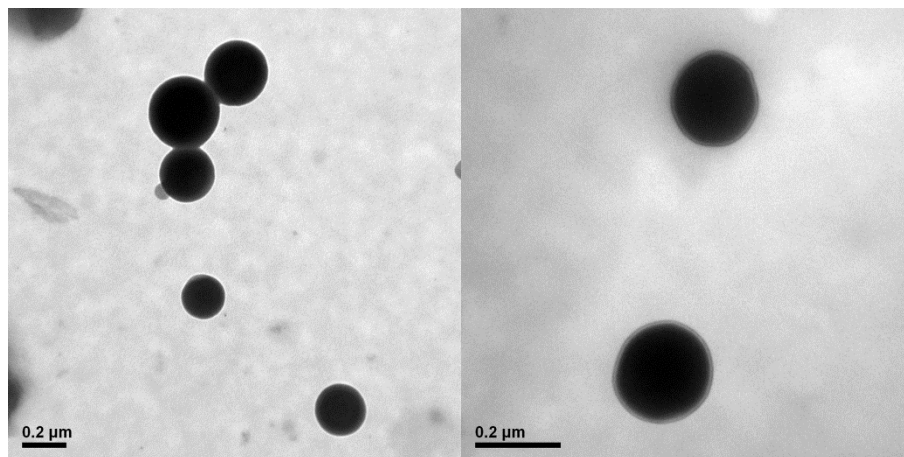


Figure S5. TEM images of *m-1* drop-cast from THF / acetone (3:7) solution.

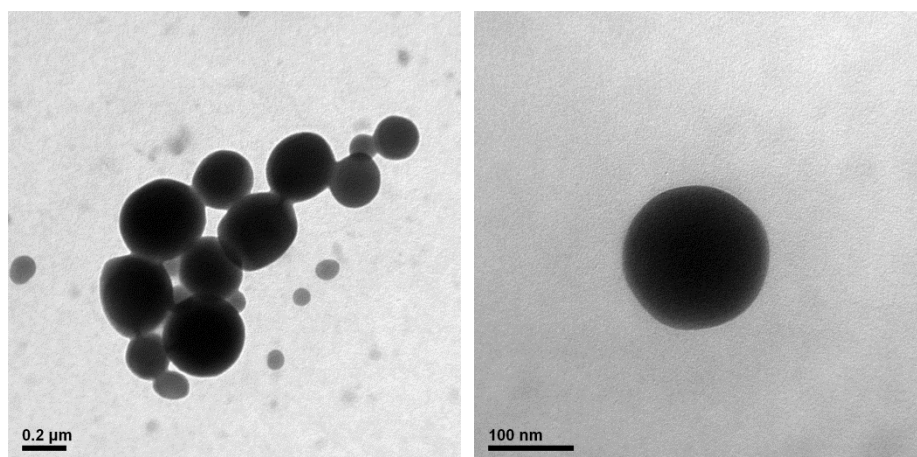


Figure S6. TEM images of *p-1* drop-cast from THF / acetone (1:9) solution.

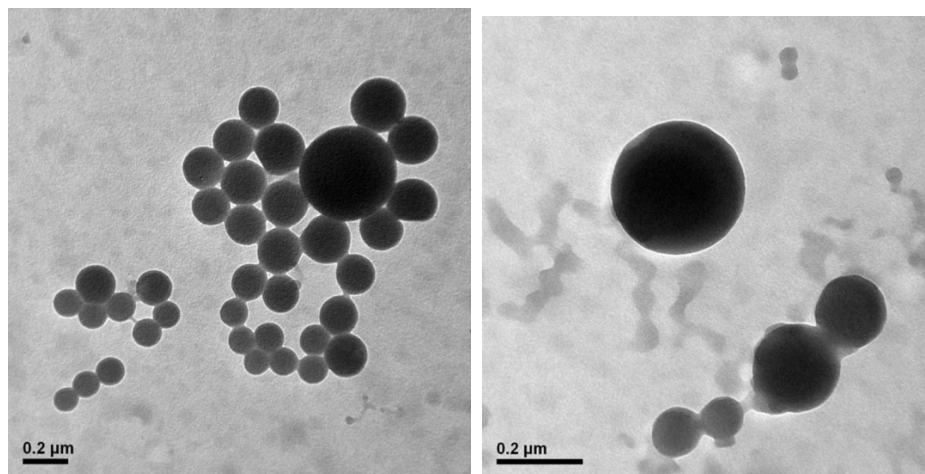


Figure S7. TEM images of *m-2* drop-cast from THF / acetone (3:7) solution.

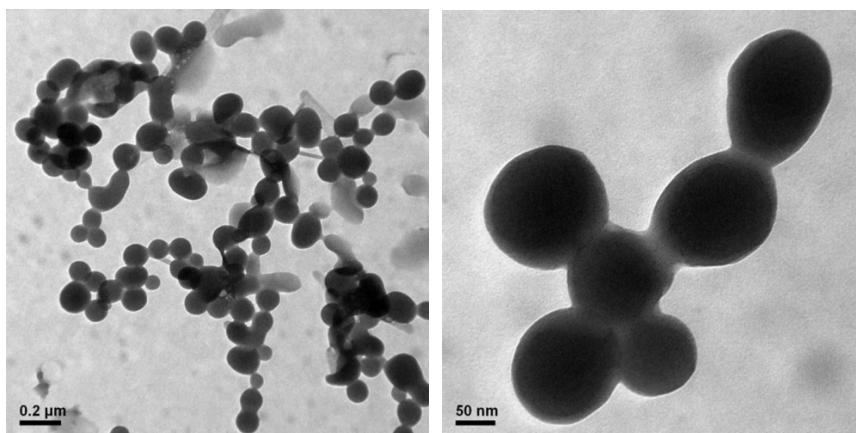


Figure S8. TEM images of *p-2* drop-cast from THF / acetone (1:9) solution.

5. Fluorescence microscopy

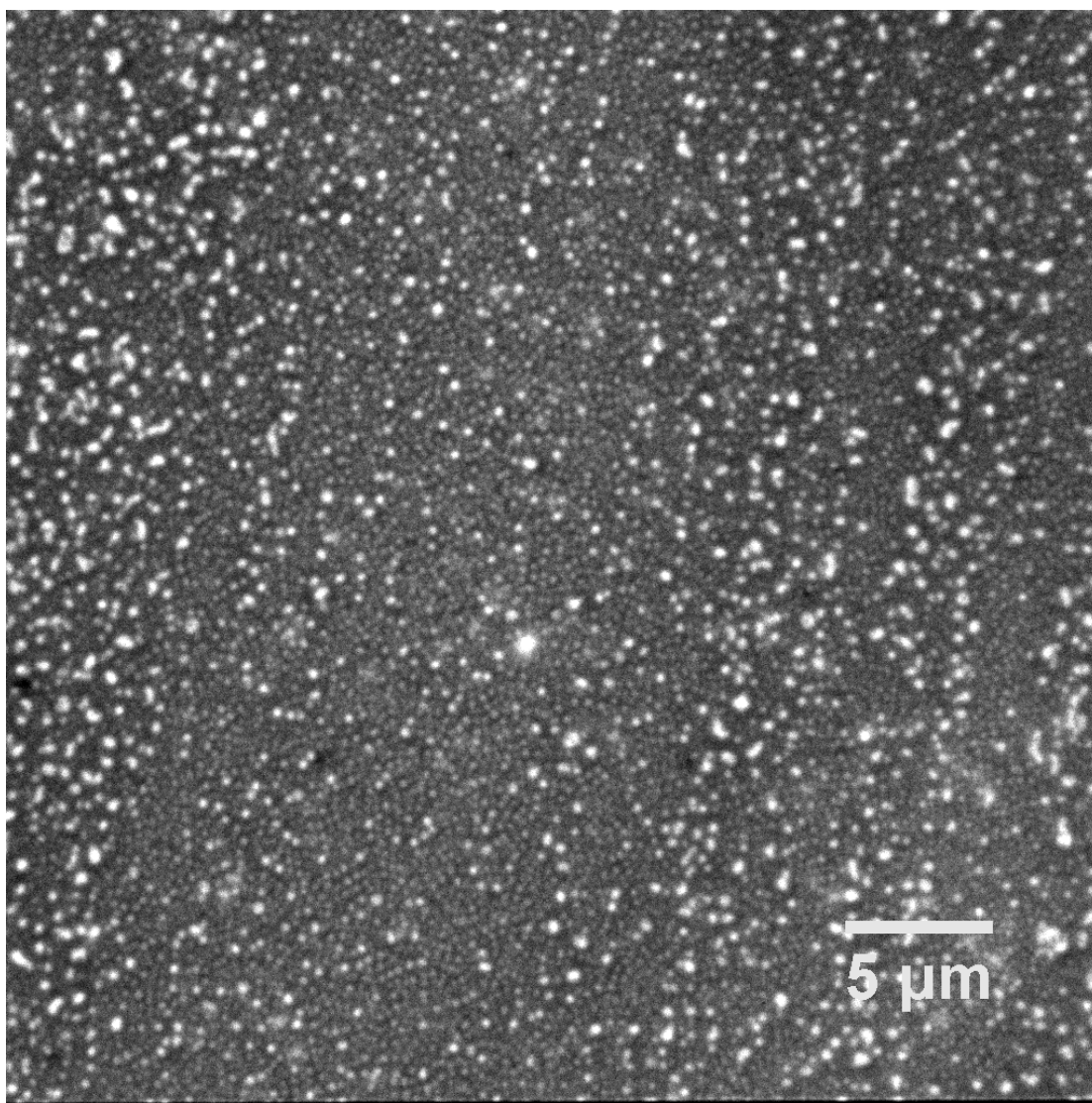


Figure S9. Wide-field fluorescence microscopy image of *m-1* deposited on a glass slide ($\lambda_{\text{ex}} = 365 \text{ nm}$, $\lambda_{\text{em}} = 390 \text{ nm}$ long pass filter).

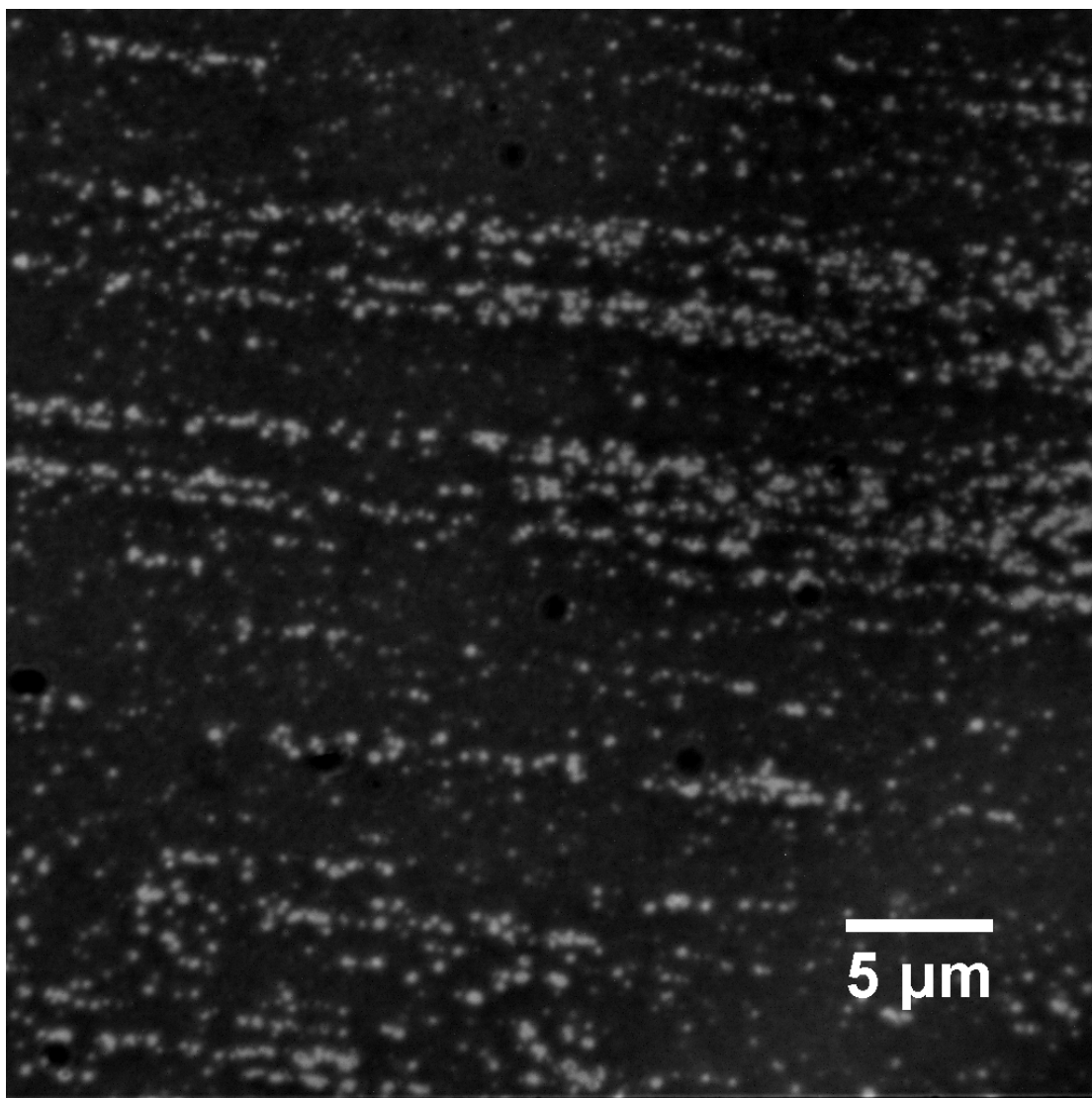


Figure S10. Wide-field fluorescence microscopy image of *p-1* deposited on a glass slide ($\lambda_{\text{ex}} = 365 \text{ nm}$, $\lambda_{\text{em}} = 390 \text{ nm}$ long pass filter).

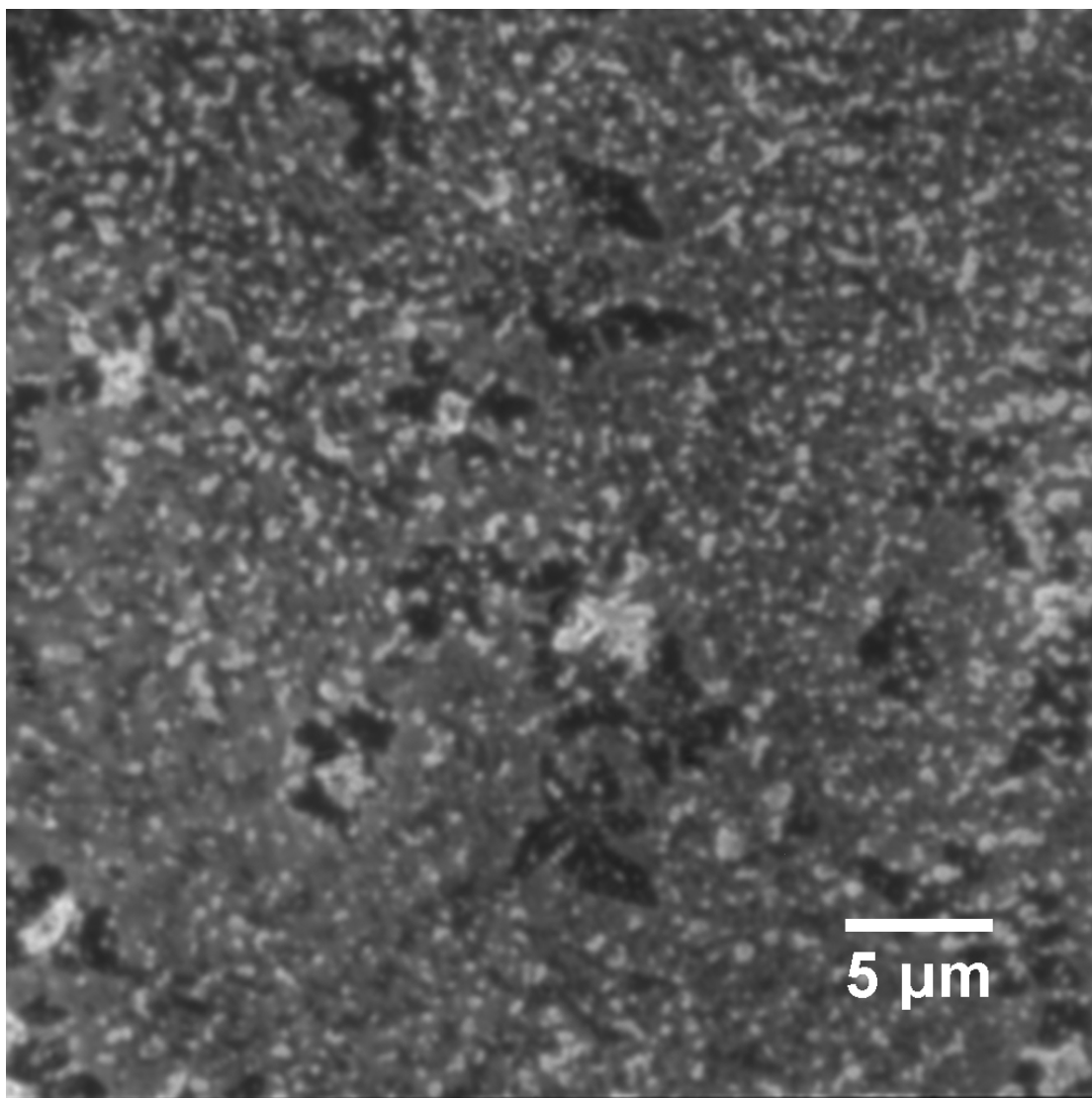


Figure S11. Wide-field fluorescence microscopy image of *m*-2 deposited on a glass slide ($\lambda_{\text{ex}} = 365$ nm, $\lambda_{\text{em}} = 390$ nm long pass filter). Scale bar is 5 μm .

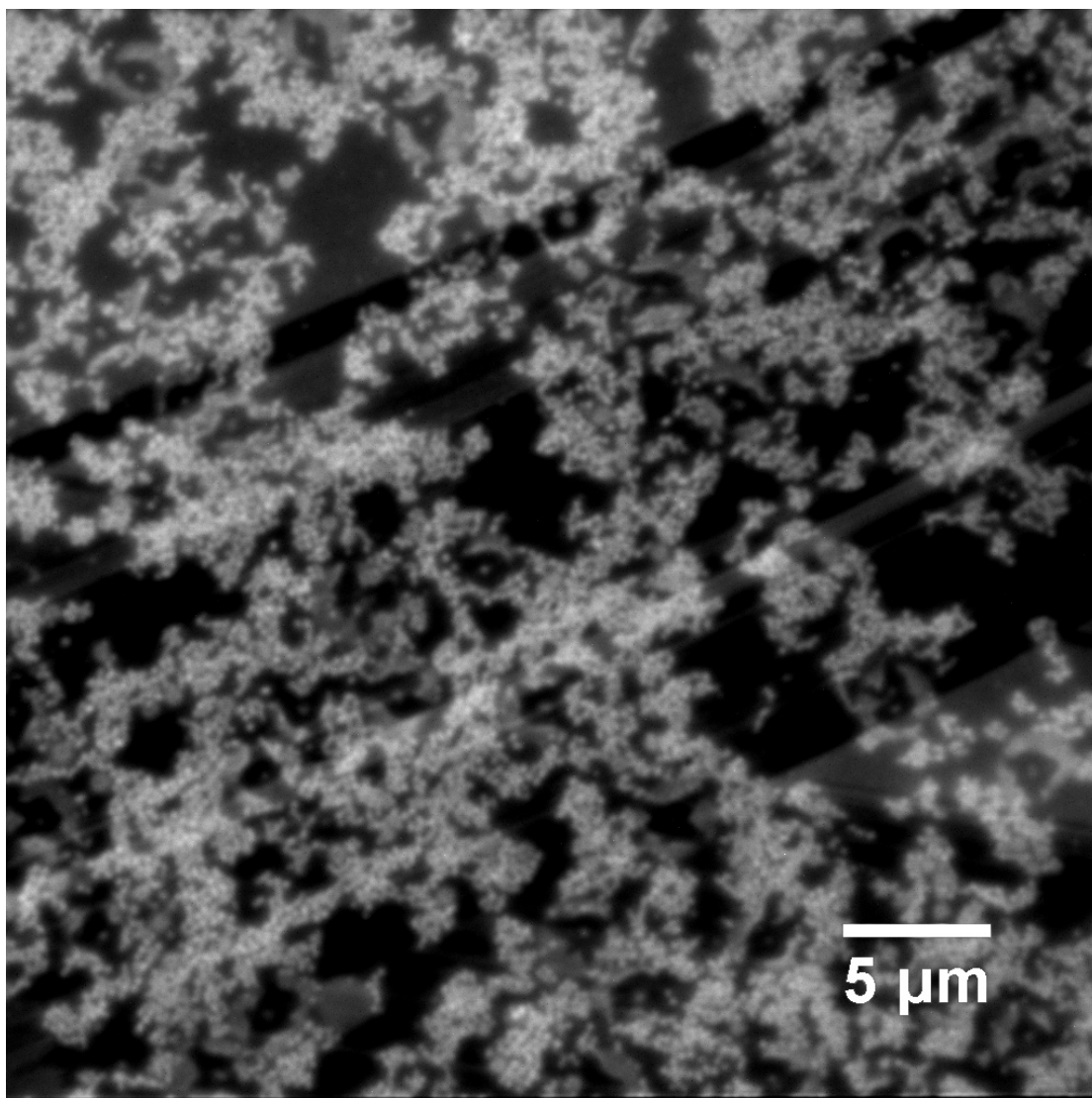


Figure S12. Wide-field fluorescence microscopy image of *p-2* deposited on a glass slide ($\lambda_{\text{ex}} = 365 \text{ nm}$, $\lambda_{\text{em}} = 390 \text{ nm}$ long pass filter). Scale bar is 5 μm .

6. Absorption and emission spectra in solution or films

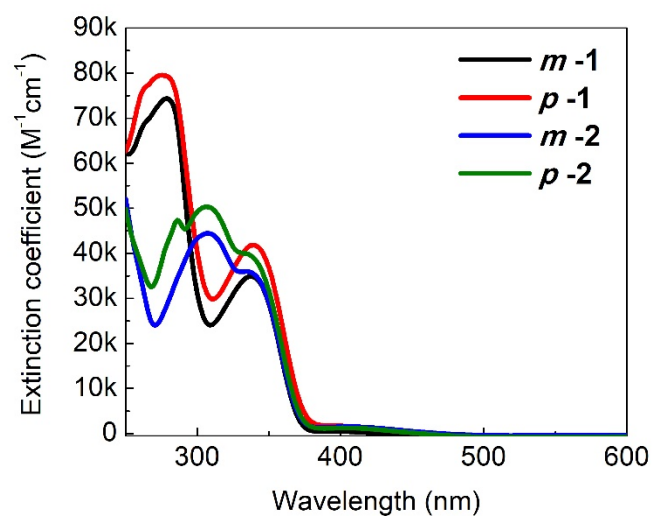


Figure S13. UV/Vis absorption spectra of **1** and **2** in THF solution (10^{-5} M).

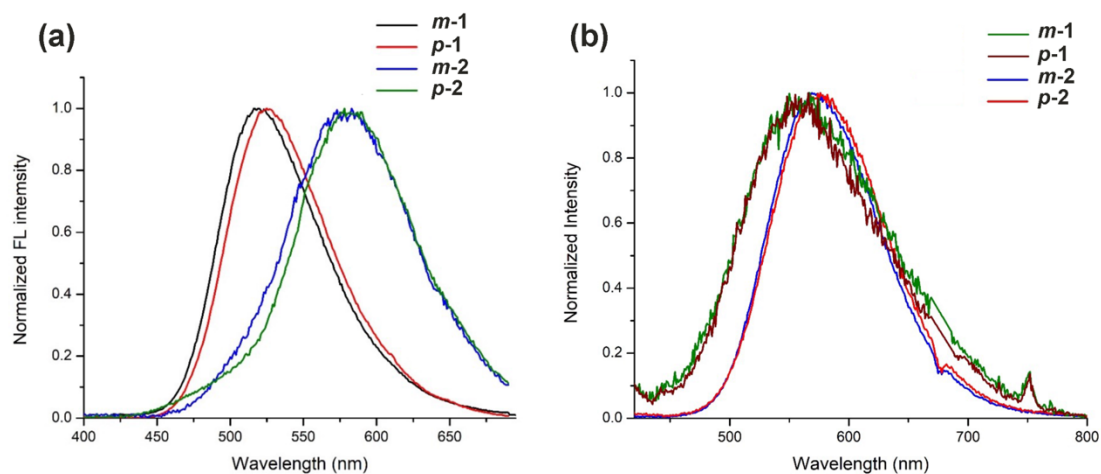


Figure S14. Fluorescence spectra of **1** and **2** in (a) toluene and (b) THF solution (10^{-5} M).

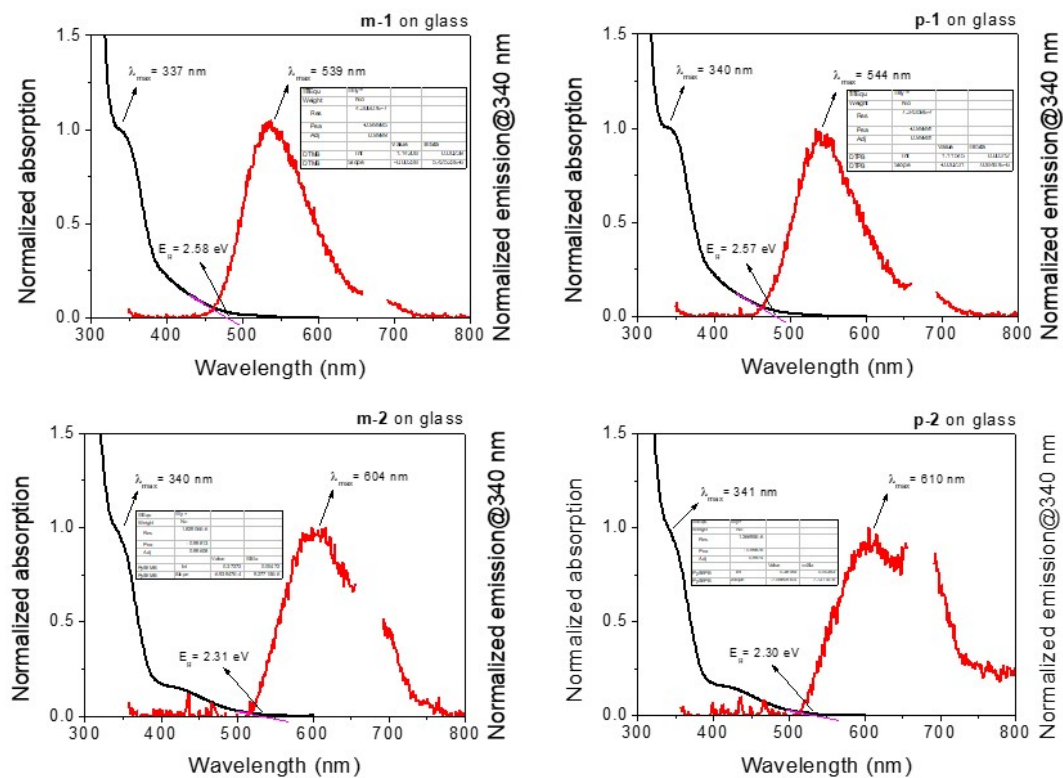


Figure S15. UV/Vis absorption and fluorescence spectra of **1** and **2** in films.

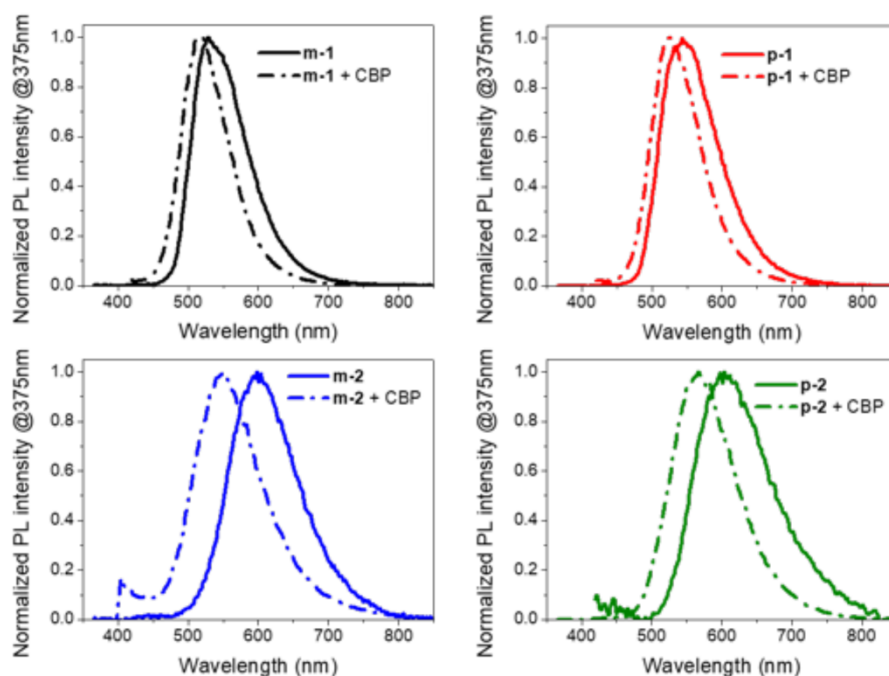


Figure S16. Photoluminescence spectra of **1** and **2** in neat and CBP films.

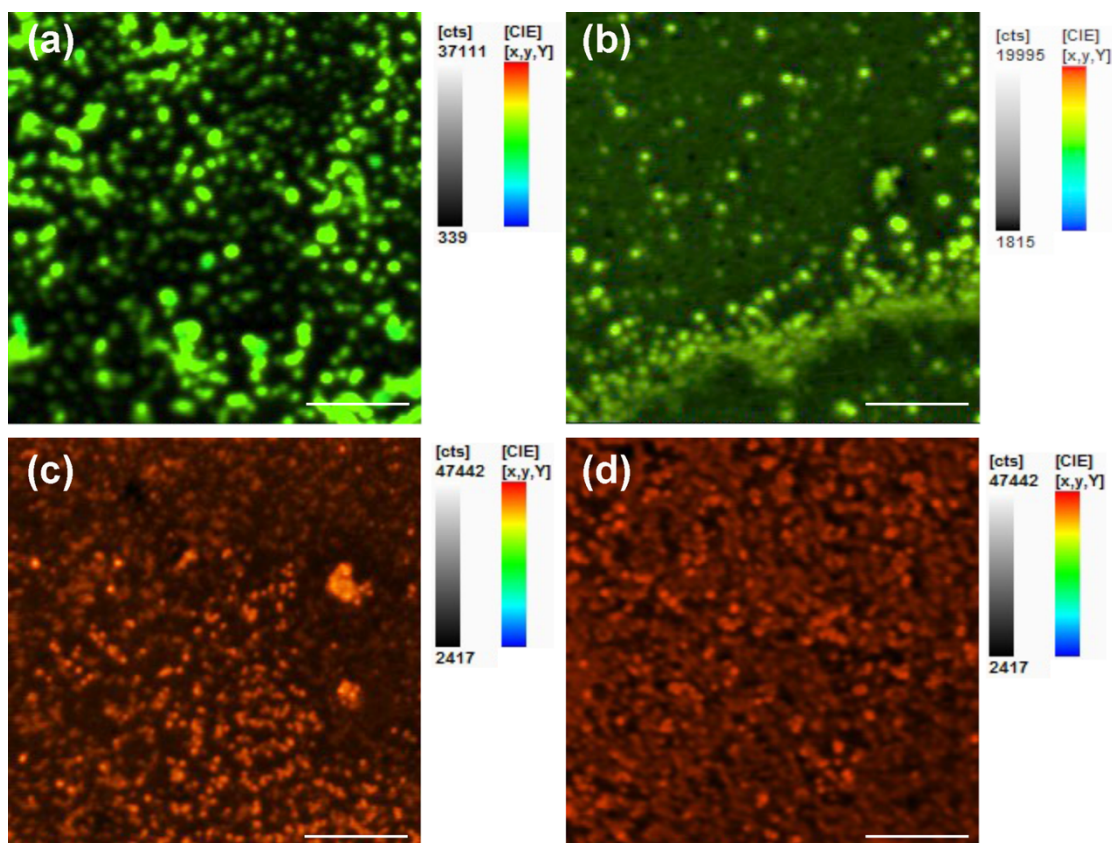


Figure S17. Real color images reconstructed from the hyperspectral images of vesicles of *m*-1 (a), *p*-1 (b), *m*-2 (c), and *p*-2 (d) deposited from acetone solutions (0.1 mM). λ_{ex} = 375 nm, scale bar is 5 μm .

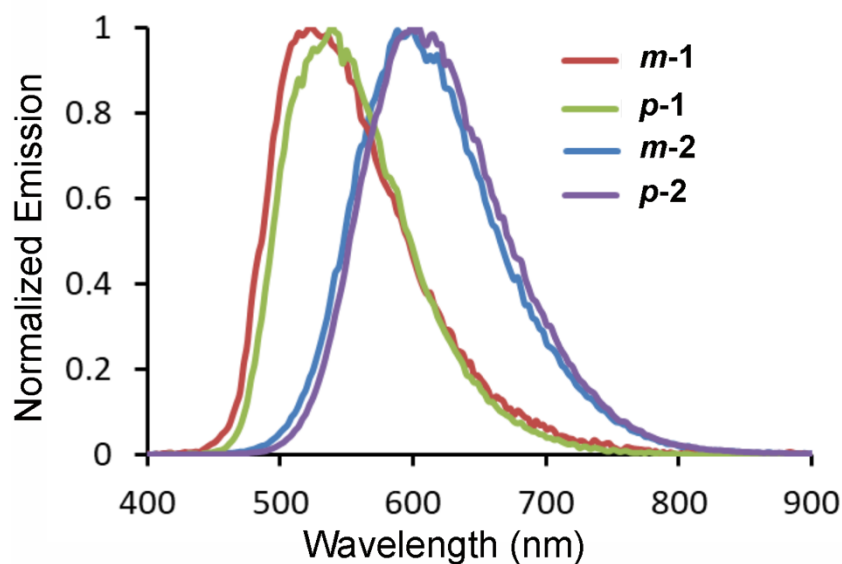


Figure S18. Photoluminescence spectra of an individual vesicle extracted from the hyperspectral images of Fig. S17 above.

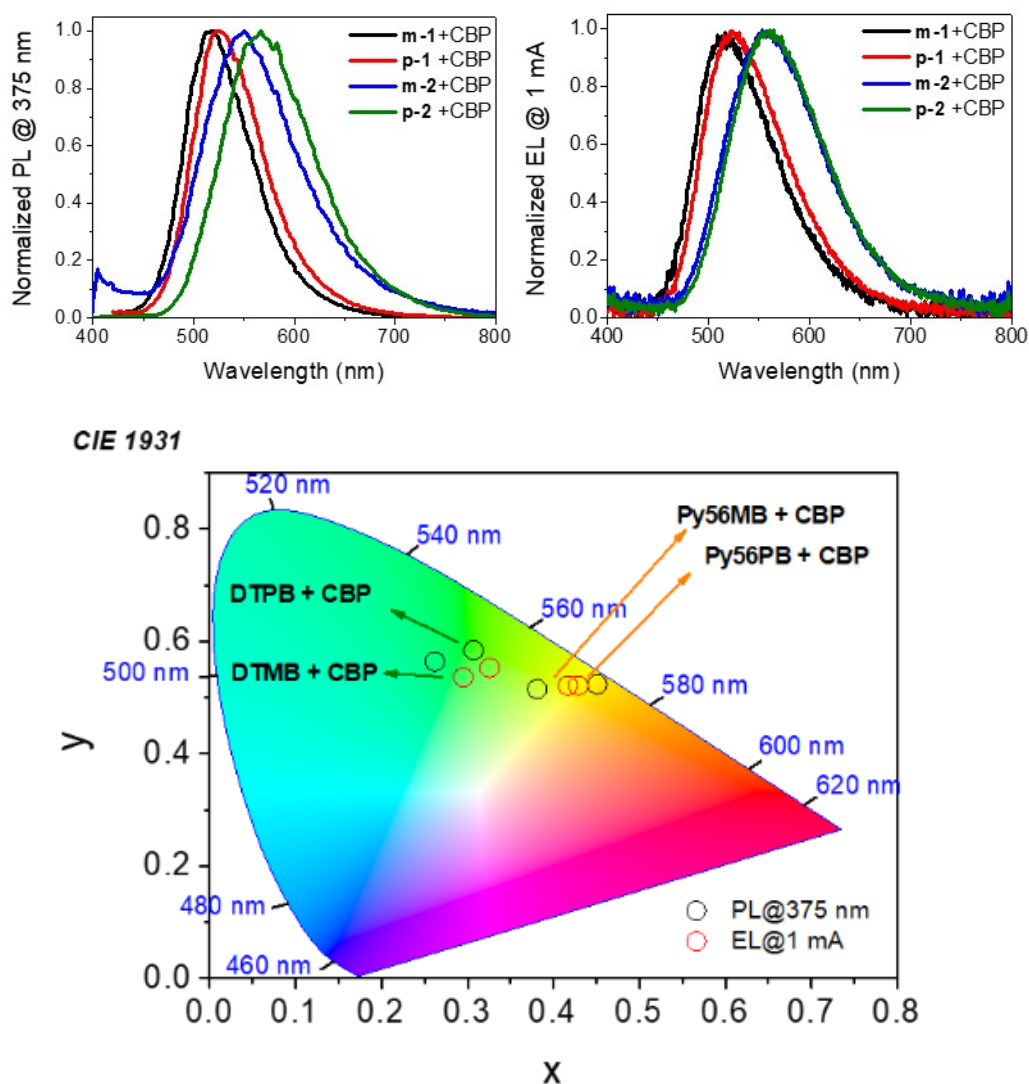


Figure S19. Comparison of the photoluminescence ($\lambda_{\text{ex}} = 375 \text{ nm}$) and electroluminescence emission (at 1 mA) of **1** and **2** doped in CBP films (top) and plotted in chromatic coordinates (bottom).

7. Solvent polarity effect on the emission spectra of *m*-1

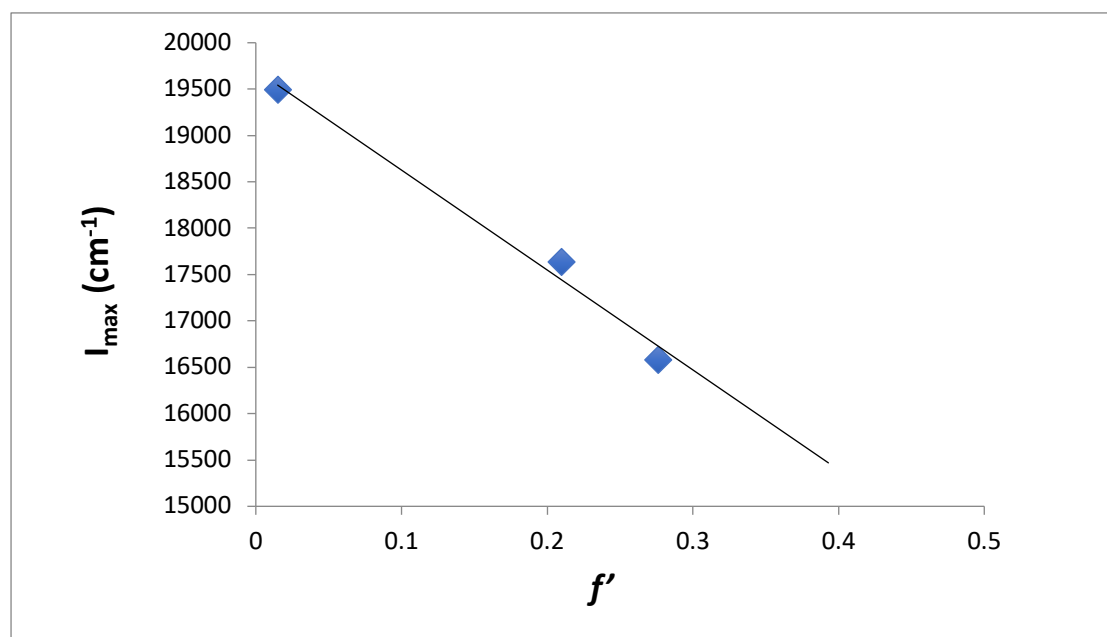


Figure S20. Lippert-Mataga plot of the variation of emission maximum of *m*-1 as a function of solvent polarity (f'). Line is best fit according to $y = -10777x + 19704$ ($r^2 = 0.986$). Assuming a solvent cavity radius of 8 Å gives $\Delta\mu = 22.8\text{D}$ corresponding to the difference in dipole moment between the ground and excited state.

8. Variable temperature fluorescence emission

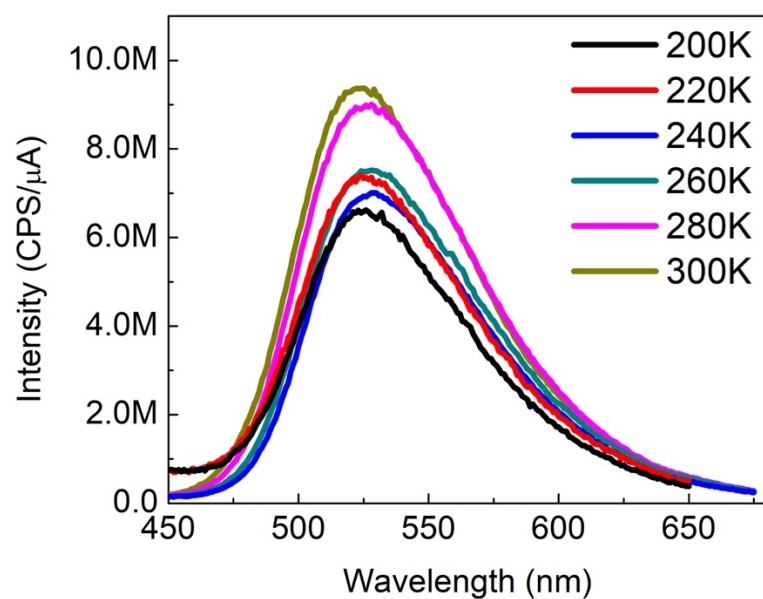


Figure S21. Fluorescence emission from *m*-1 in degassed toluene solution ($\lambda_{\text{ex}} = 350$ nm) as a function of temperature.

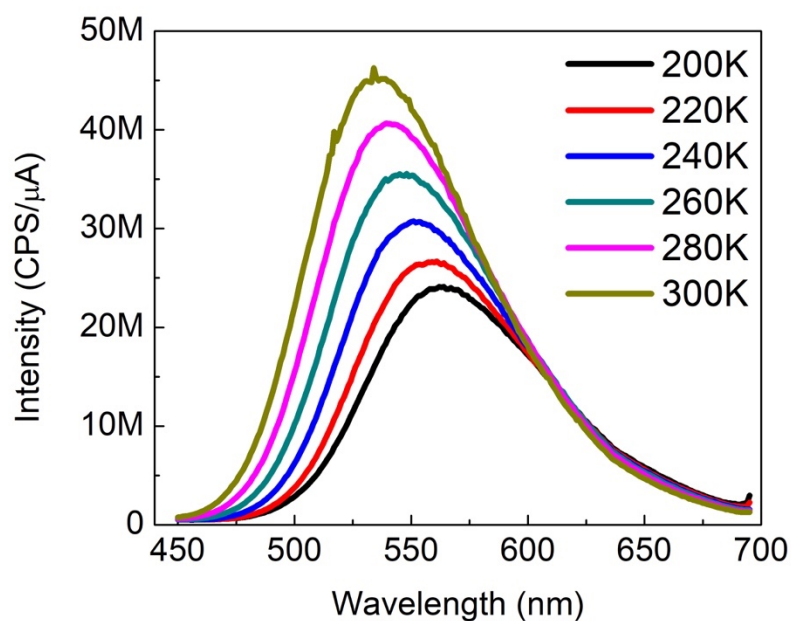


Figure S22. Fluorescence emission from *p*-1 in degassed toluene solution ($\lambda_{\text{ex}} = 350$ nm) as a function of temperature.

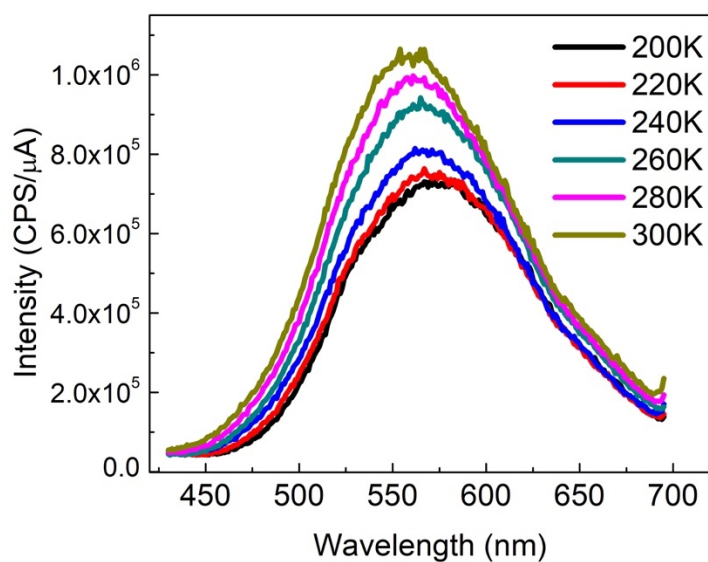


Figure S23. Fluorescence emission from *m*-2 in degassed THF solution ($\lambda_{\text{ex}} = 350$ nm) as a function of temperature.

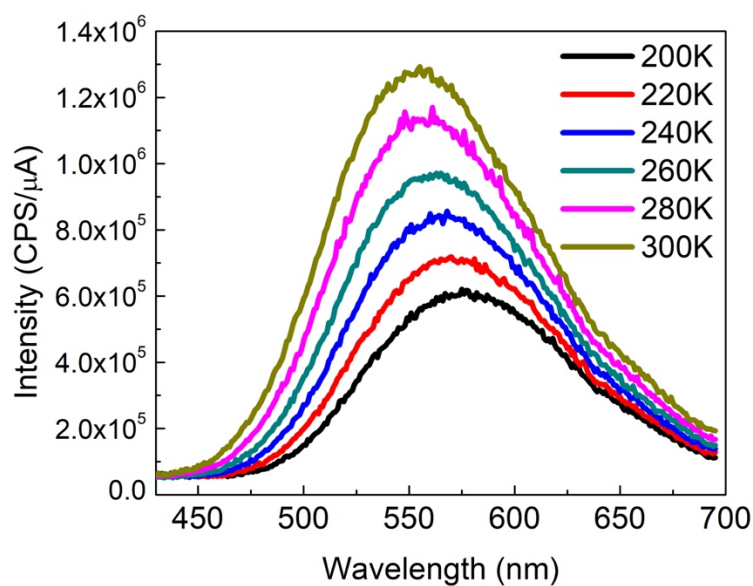


Figure S24. Fluorescence emission from *p-2* in degassed THD solution ($\lambda_{\text{ex}} = 350$ nm) as a function of temperature.

9. Variable temperature emission decays

Table S1. Deconvoluted decay parameters for *m-1* in degassed toluene solutions ($\lambda_{\text{ex}} = 310$ nm).

T	A1	t1	A2	t2	DF	PF
300	0.0779	1400	5.6	25.9	0.43	0.57
292	0.1722	1500	12.6	26.37	0.44	0.56
280	0.0933	1474	7.5	26.13	0.41	0.59
260	0.0629	1661	5.3	29.3	0.40	0.60
240	0.0545	1849	5.61	29.3	0.38	0.62
220	0.0562	2029	8	29.6	0.33	0.67
200	0.0538	1982	4.81	33.7		

Table S2. Deconvoluted decay parameters for *p*-1 in degassed toluene solutions ($\lambda_{\text{ex}} = 310 \text{ nm}$).

T	A1	t1	A2	t2	DF	PF
300	6.24	27.12	0.1432	1510	0.56	0.44
280	8.6	29.6	0.18	1560	0.52	0.48
260	10.34	29.719	0.1405	1576	0.42	0.58
240	4.88	31.7	0.133	1436	0.55	0.45
220	7.31	31.72	0.34	1439	0.68	0.32
200	6.62	32.4	0.35	1342	0.69	0.31

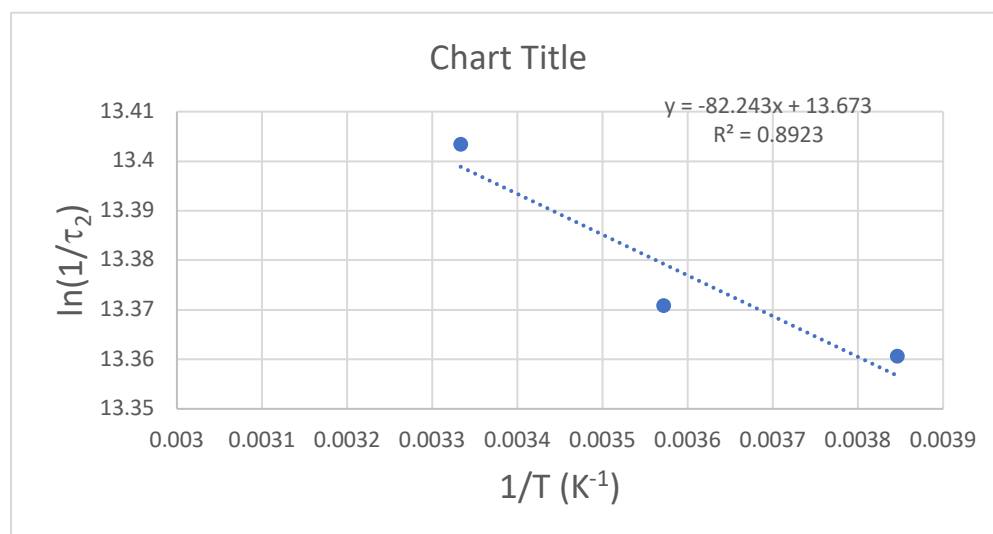


Figure S25. Arrhenius plot of the reciprocal of the long-lived decay component for *p*-1. Data is truncated as no further increase in lifetime is observed at lower temperatures.

Table S3. Deconvoluted decay parameters for *m*-2 in degassed toluene solutions ($\lambda_{\text{ex}} = 310 \text{ nm}$).

T	A1	T1	A2	T2	A3	T3
300	5.15	5.03	0.0283	1922	8.5	18.19
280	4.27	5.78	0.026	1969	8.95	18.59
260	3.66	5.2	0.0215	2076	9.2	18.67
240	4.03	4.41	0.0198	2690	9.1	19.11
220	4.64	5.52	0.018	3133	8.33	20.03
200	4.44	7.04	0.0112	3411	8.08	21.22

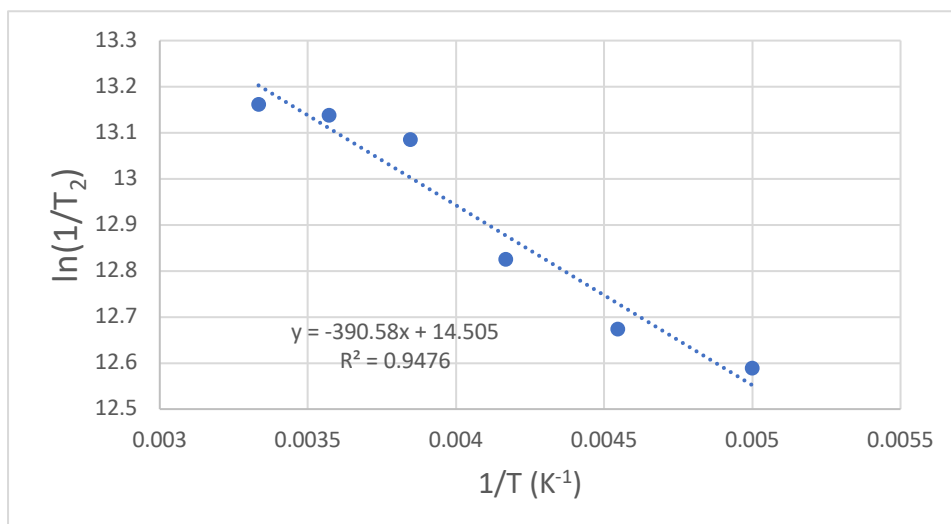


Figure S26. Arrhenius plot of the reciprocal of the long-lived decay component for *p*-1.

Table S4. Deconvoluted decay parameters for *p*-2 in degassed toluene solutions ($\lambda_{ex} = 310$ nm).

T	A1	T1	A2	T2	A3	T3
300	3.1	4.46	0.0204	1397	10.94	14.54
280	3.42	4.9	0.018	1584	10.48	14.957
260	3.61	5.65	0.0119	2086	10.14	15.28
240	4.73	5.84	0.0097	2503	9.2	16.1
220	10.6	2.8	0.008	3322	10.3	16.06
200	6.54	6.1	0.0074	3727	6.97	18.48

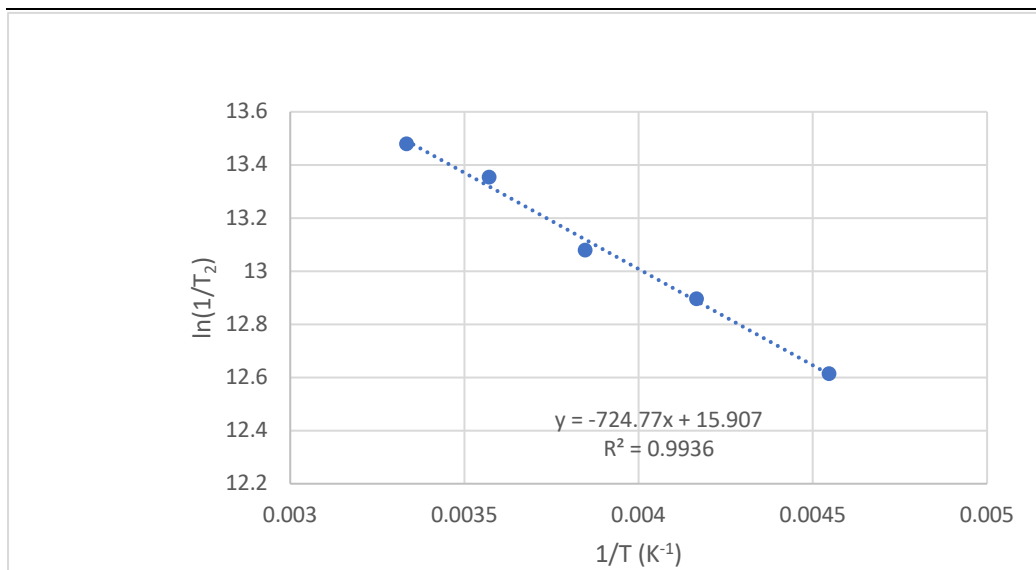


Figure S27. Arrhenius plot of the reciprocal of the long-lived decay component for *p*-2.

10. Redox properties and energy levels of 1 and 2

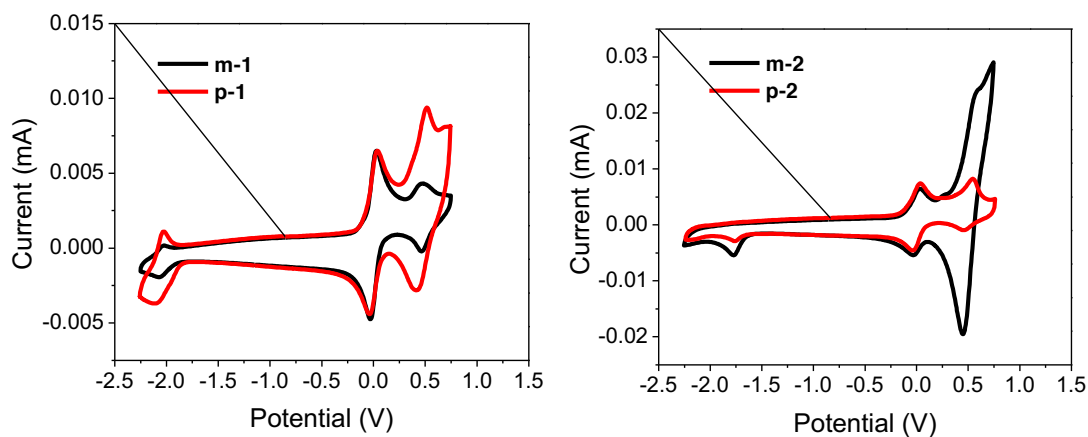


Figure S28. Cyclic voltammograms of **1** and **2** in acetonitrile (1.0 mM). (vs. Fc/Fc⁺, Fc/Fc⁺ = ferrocene/ferrocenium).

Table S5. Summary of energy levels of **1** and **2**.

Compound	HOMO ^a (eV)	LUMO ^a (eV)	Electrochemical ^b E_g on film (eV)	Optical E_g^c on film (eV)	Optical E_g^c in solution (eV)
m-1	-5.60	-3.05	2.55	2.58	2.60
p-1	-5.58	-3.04	2.55	2.57	2.56
m-2	-5.61	-3.32	2.29	2.31	2.51
p-2	-5.59	-3.34	2.26	2.30	2.51

^a $E_{\text{HOMO}} = - (5.10 + E_{\text{onset}}^{\text{ox}})$ eV; $E_{\text{LUMO}} = - (5.10 + E_{\text{onset}}^{\text{red}})$ eV. ^bElectrochemical gaps calculated according to the equation $E_g = (E_{\text{LUMO}} - E_{\text{HOMO}})$ eV. ^cOptical gaps calculated according to the onset absorption.

11. ^1H NMR and ^{13}C NMR spectra

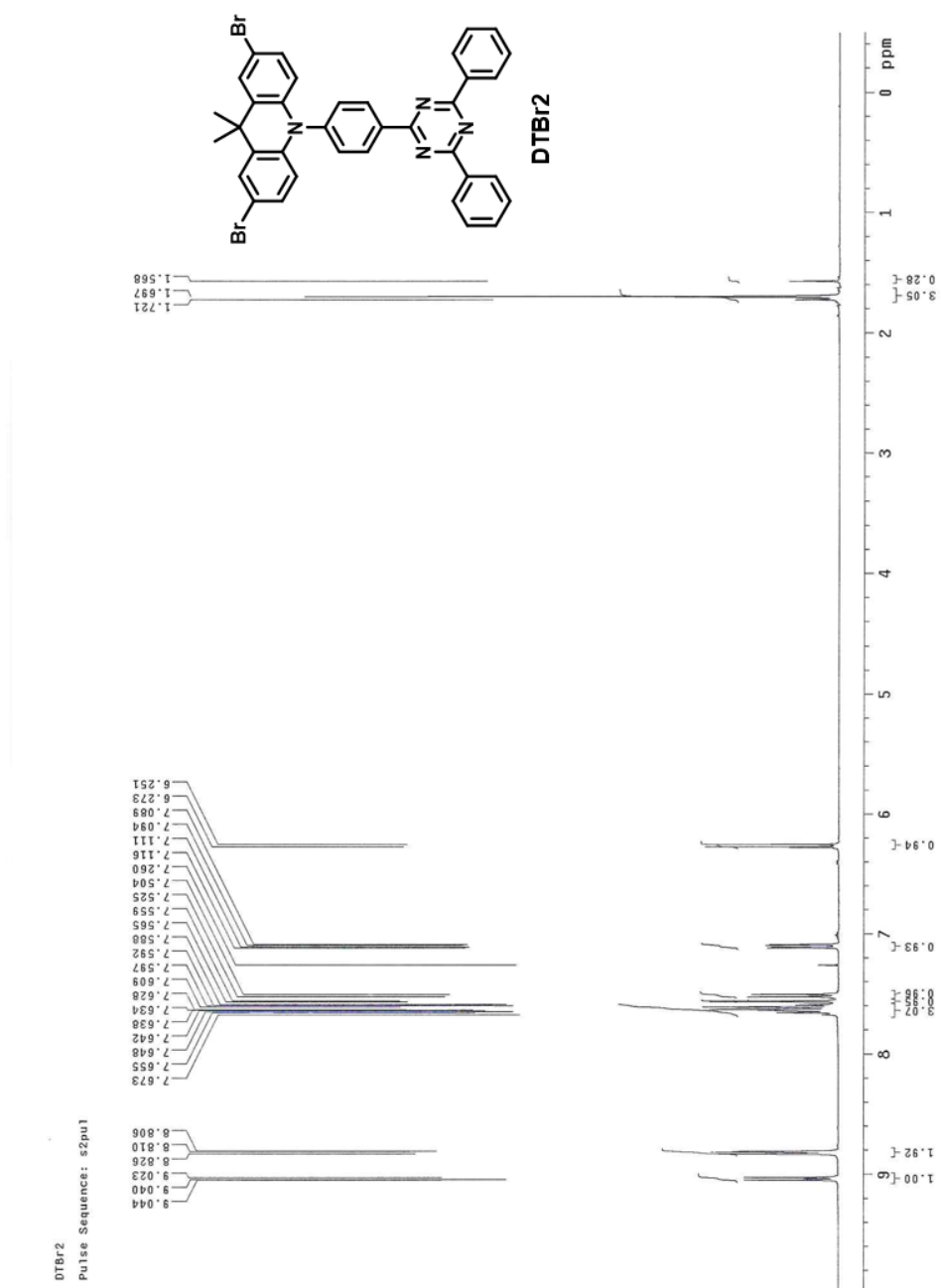


Figure S29. ^1H NMR spectrum of DTBr2.

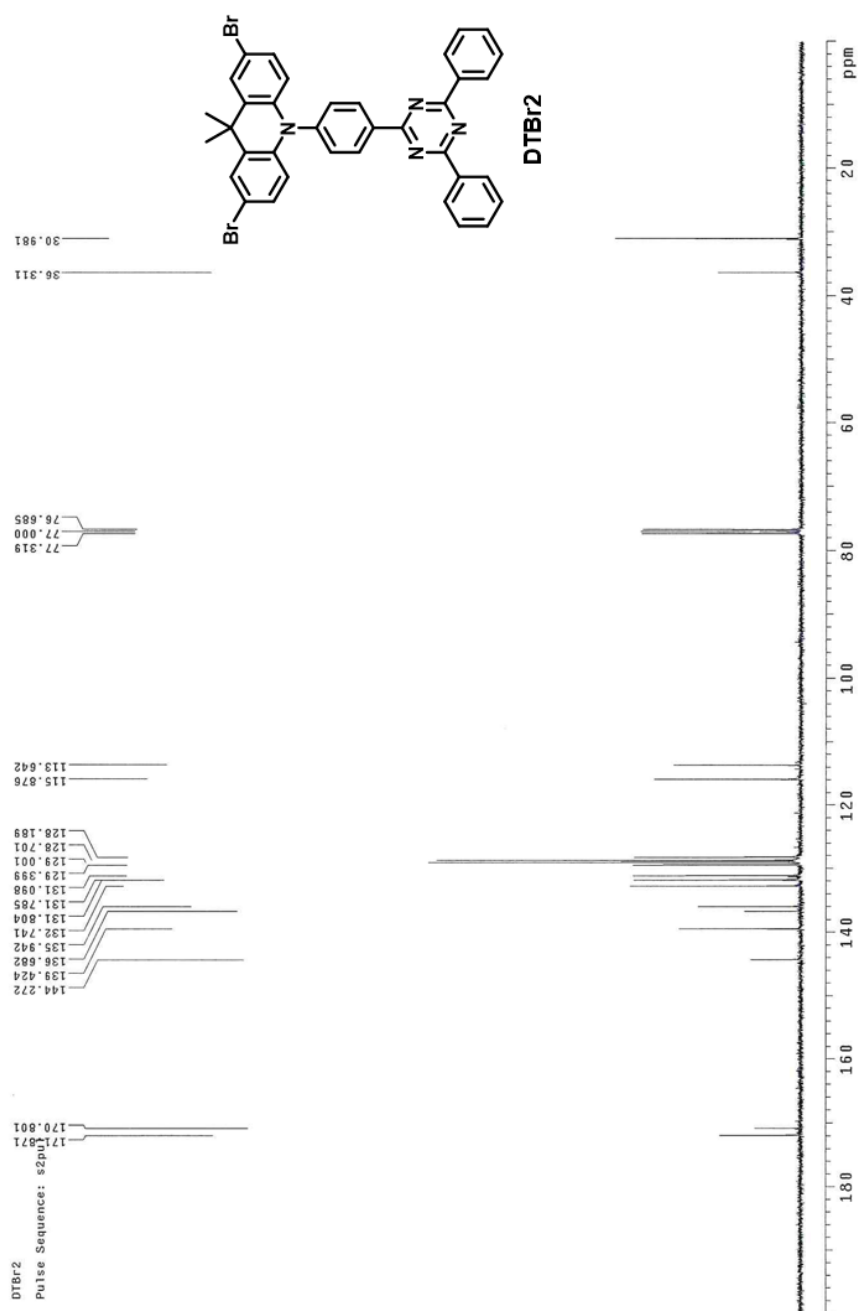


Figure S30. ¹³C NMR spectrum of DTBr2.

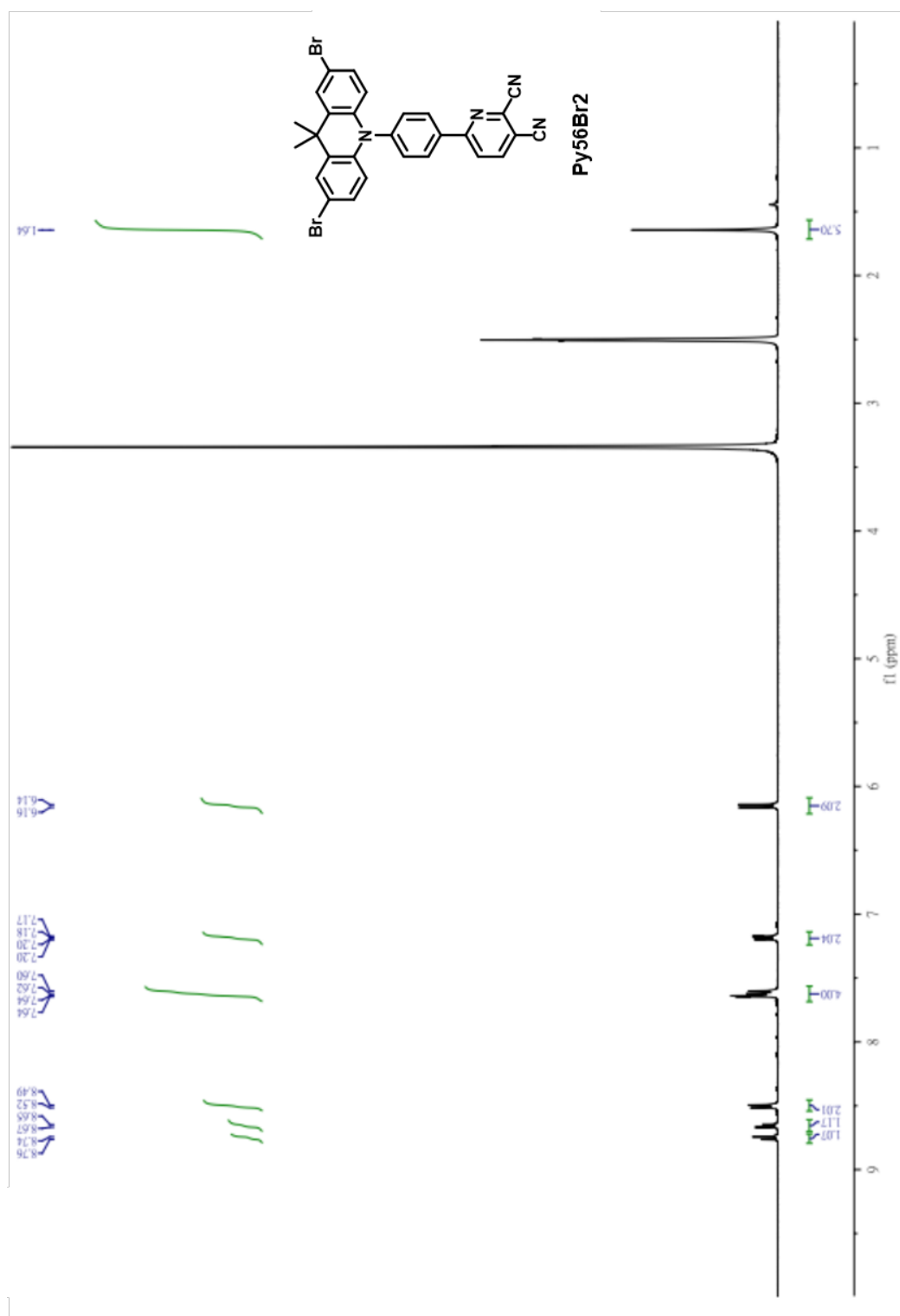


Figure S31. ¹H NMR spectrum of **Py56Br2**.

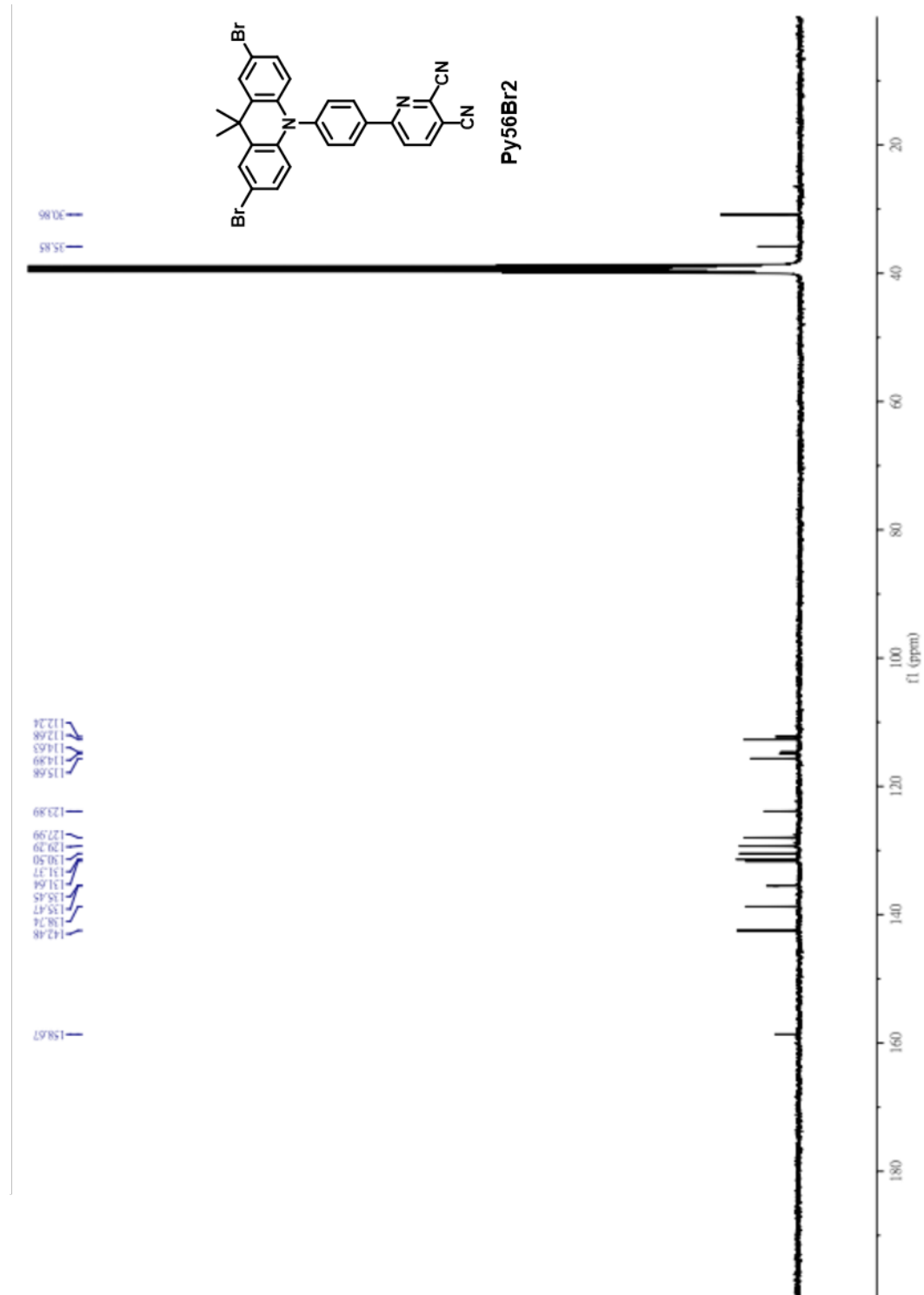


Figure S32. ¹³C NMR spectrum of **Py56Br2**.

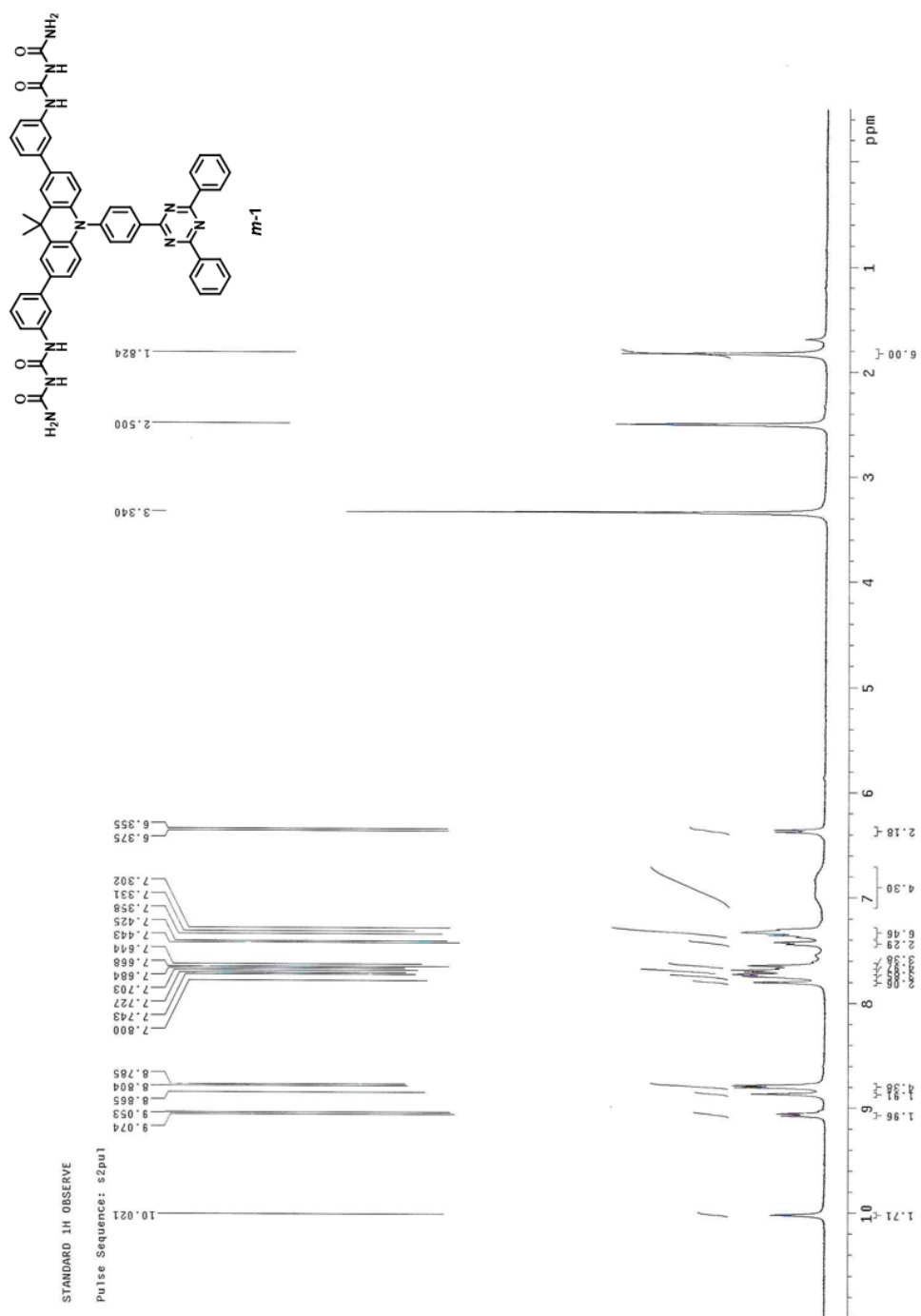


Figure S33 ¹H NMR spectrum of *m-1*.

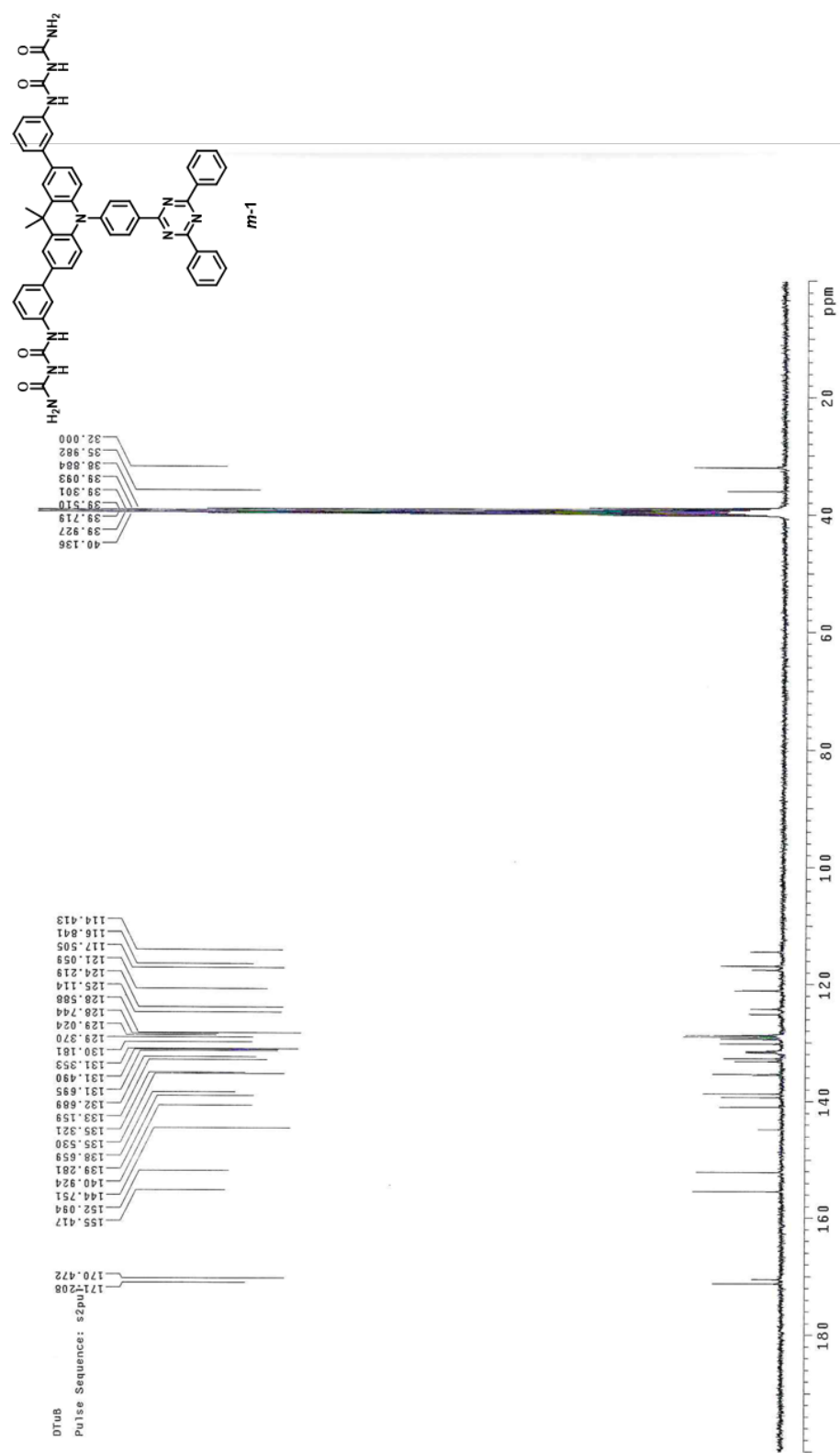


Figure S34. ¹³C NMR spectrum of *m-1*.

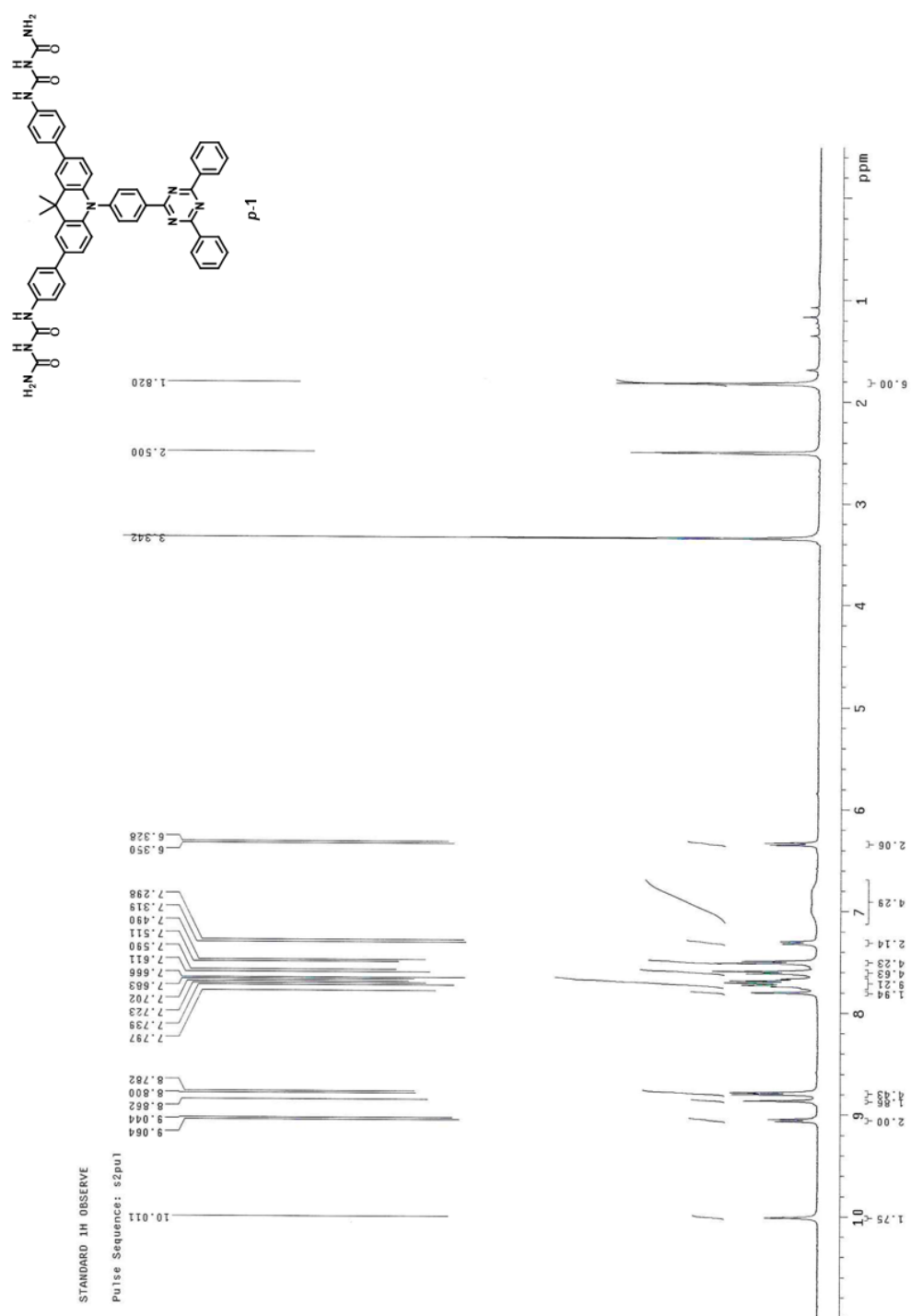


Figure S35. ^1H NMR spectrum of **p-1**.

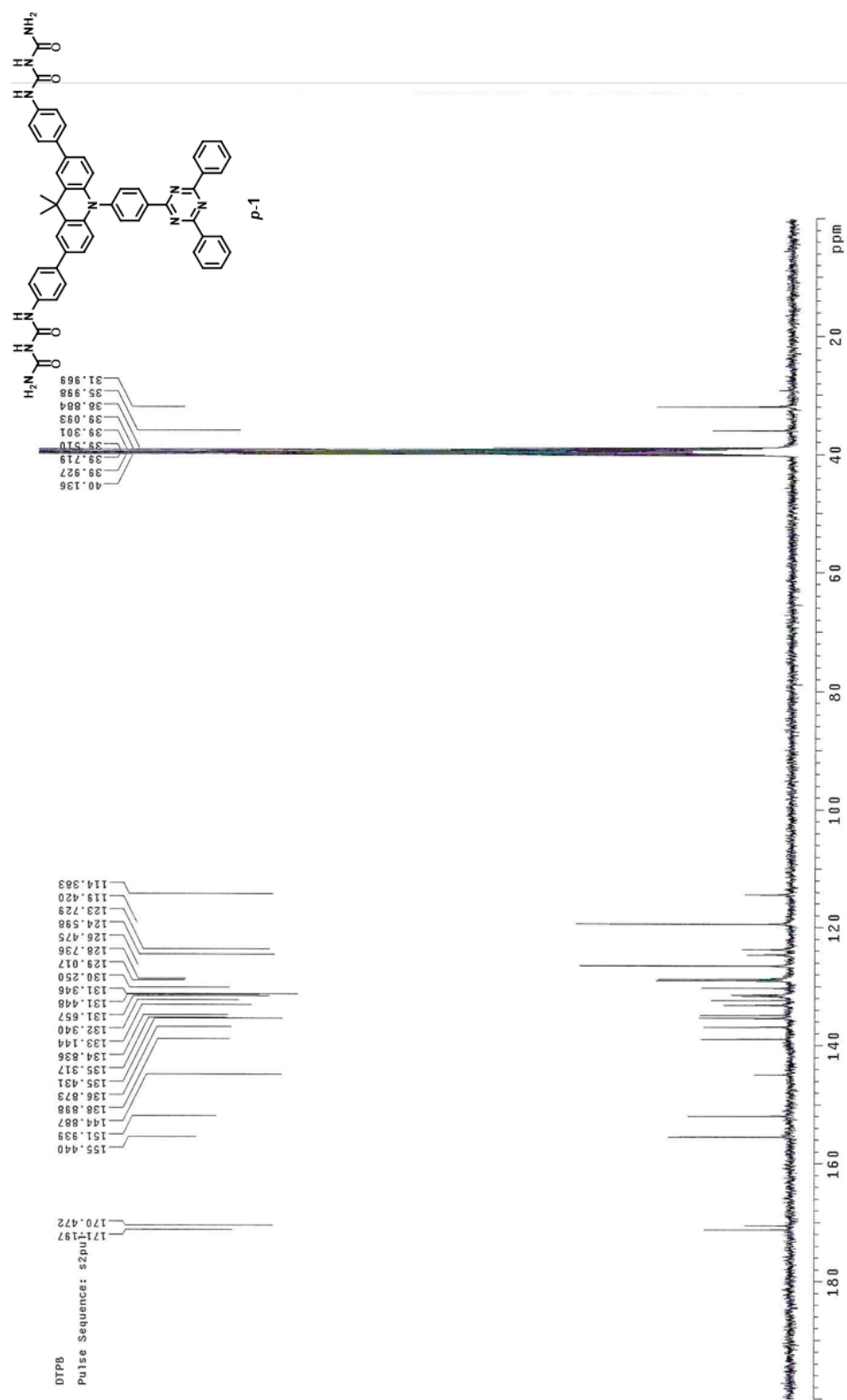


Figure S36. ¹³C NMR spectrum of *p*-1.

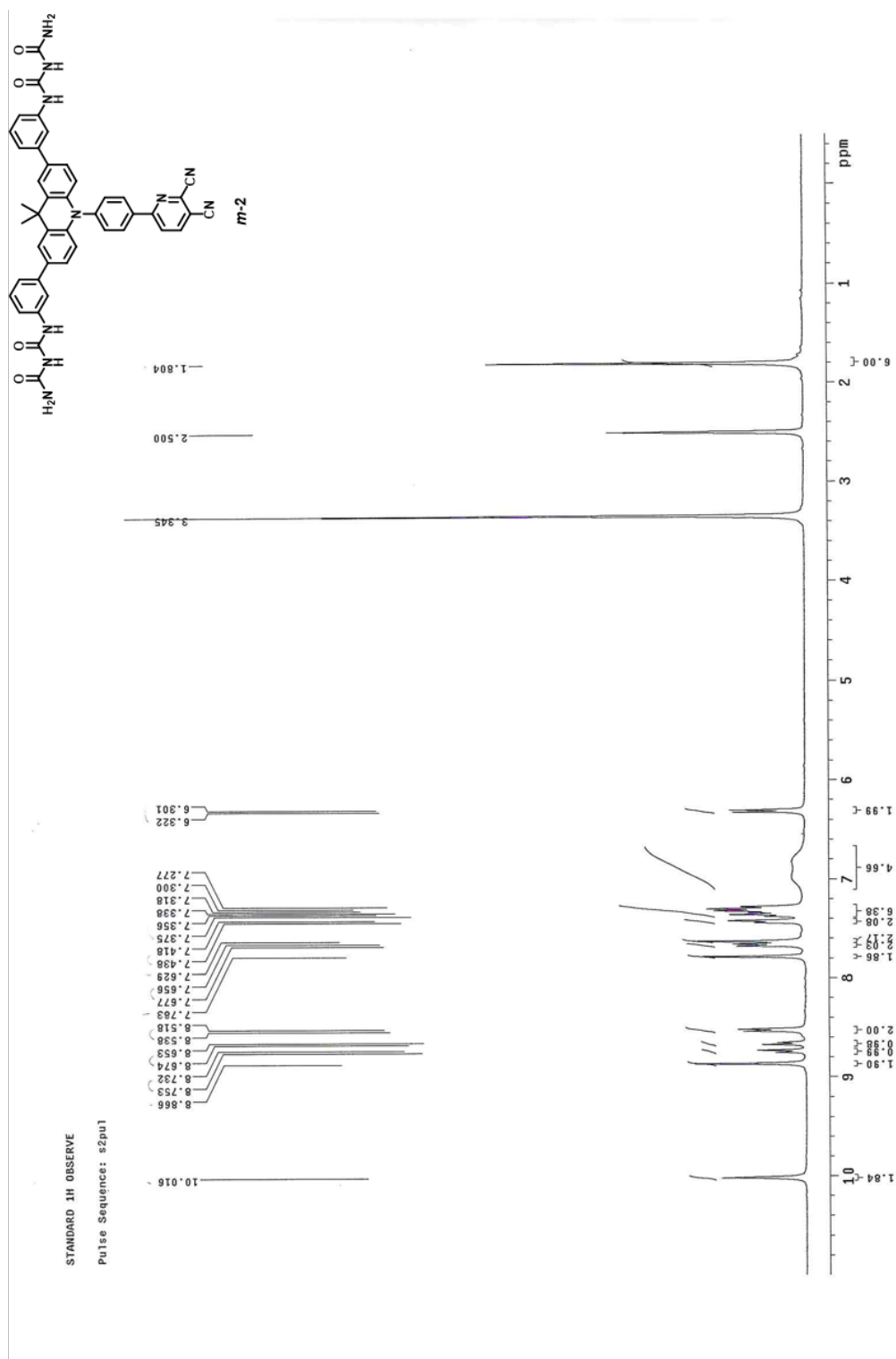


Figure S37. ¹H NMR spectrum of *m-2*.

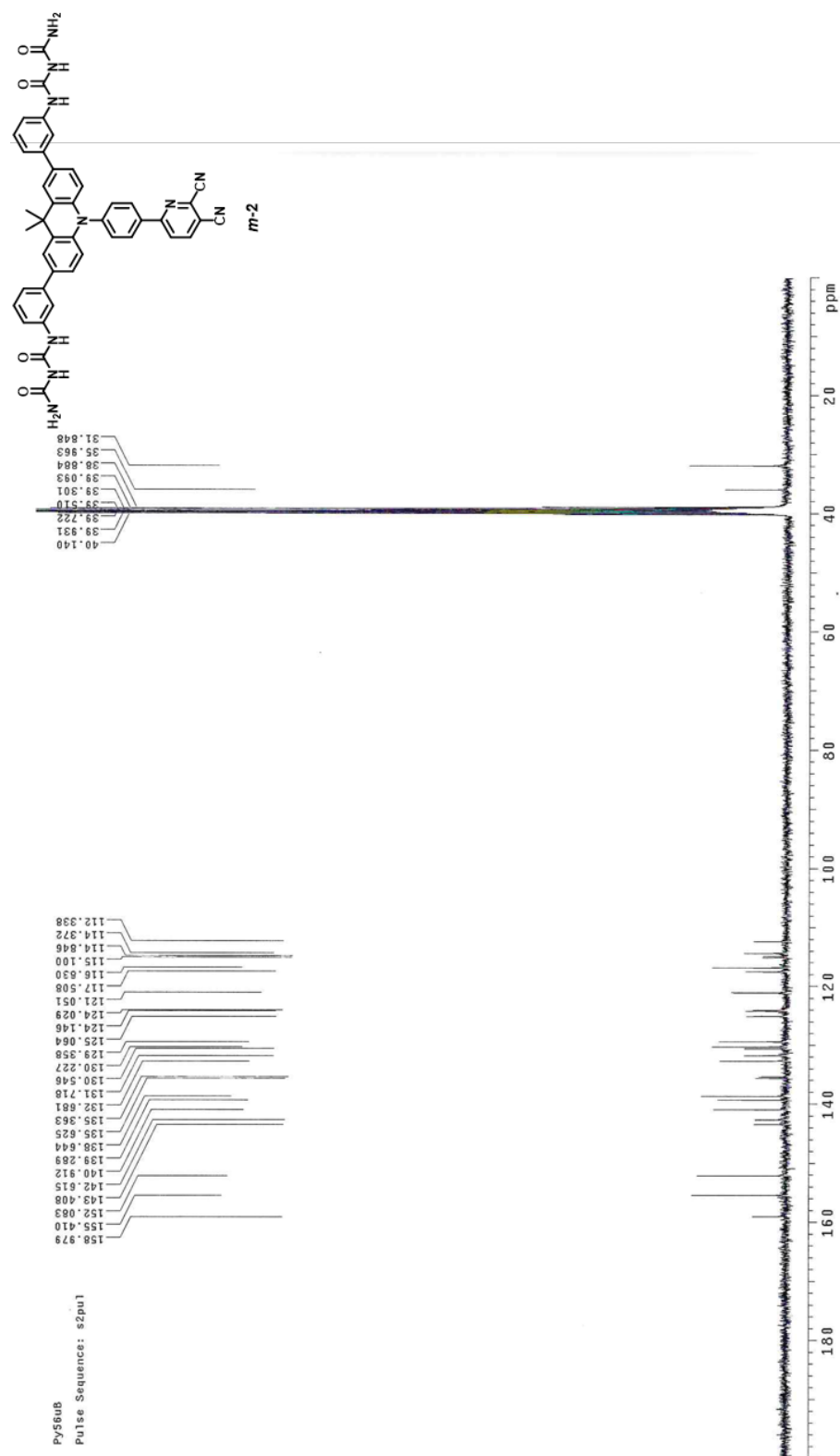


Figure S38. ^{13}C NMR spectrum of *m-2*.

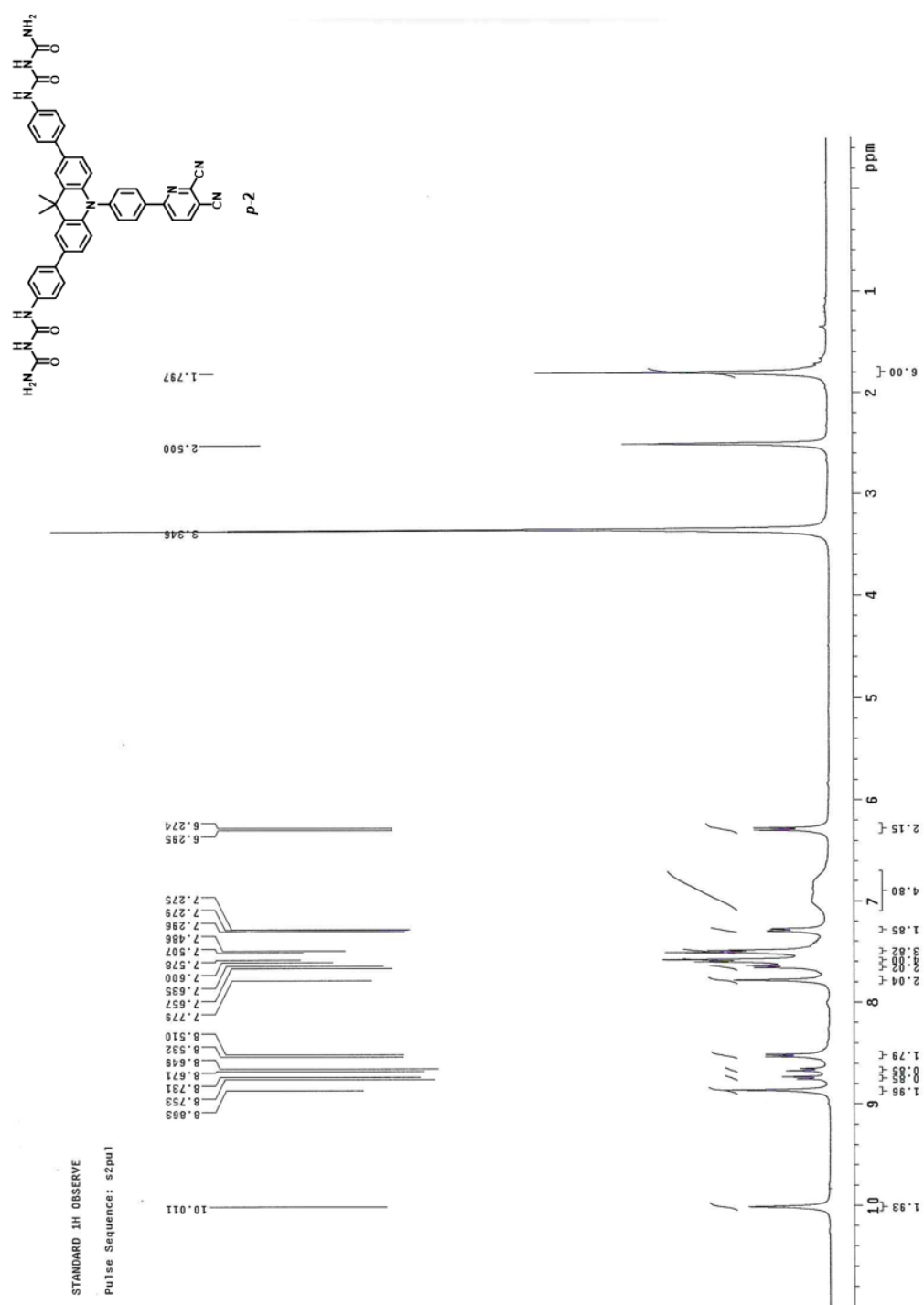


Figure S39. ^1H NMR spectrum of **p-2**.

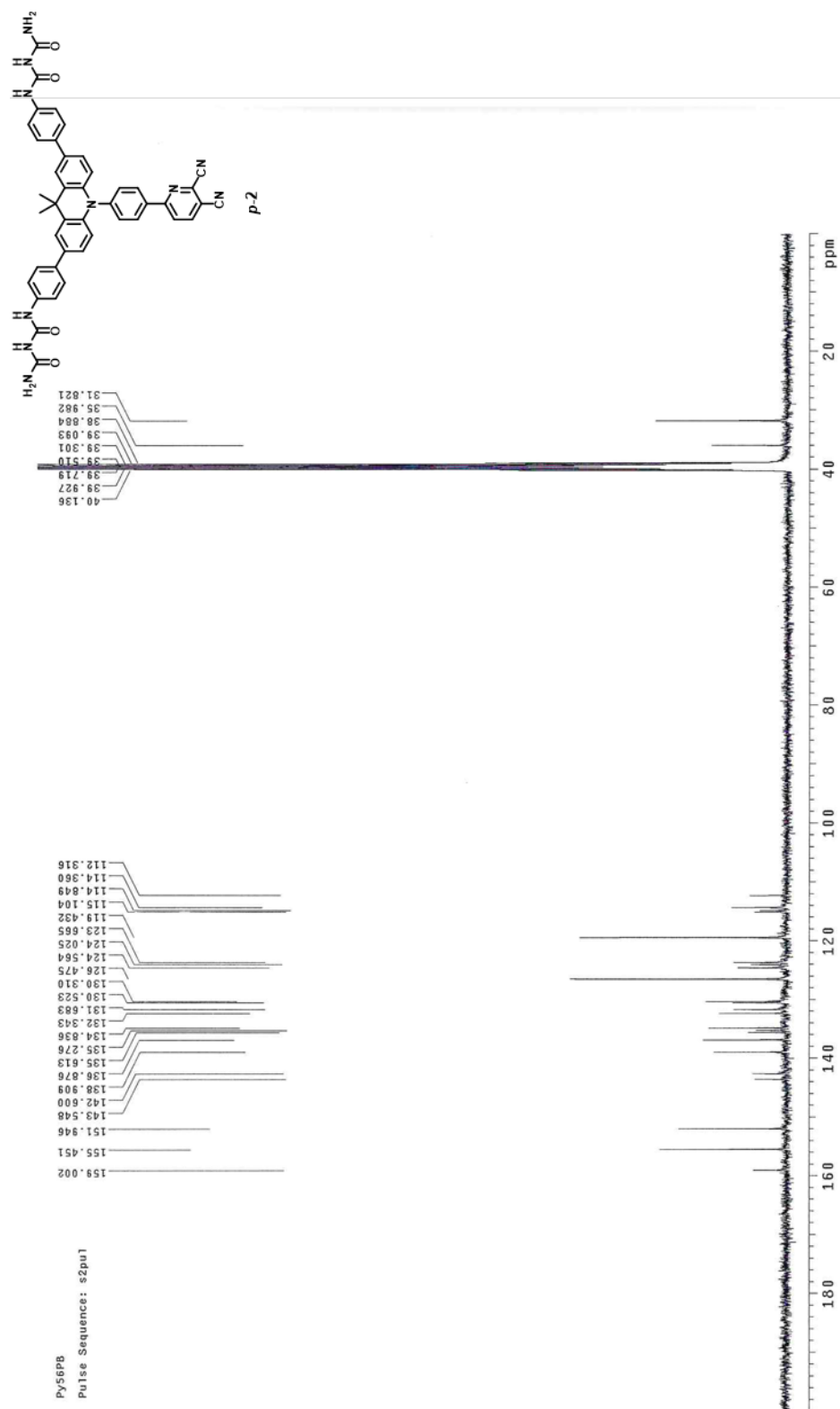


Figure S40. ^{13}C NMR spectrum of *p-2*.